

**DEVELOPMENT OF ALKALI-ACTIVATED BINDERS
UTILIZING INDIGENOUS INDUSTRIAL WASTE
MATERIALS**

BY

SAMI SALEM HAMEED

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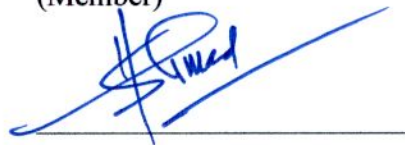
This thesis, written by **SAMI SALEM HAMEED** under the direction his thesis advisor and approved by his thesis committee, has been presented and accepted by the Dean of Graduate Studies, in partial fulfillment of the requirements for the degree of **MASTER OF SCIENCE IN CIVIL ENGINEERING**.



Dr. Salah U. Al-Dulaijan
(Advisor)



Dr. Mohammed Maslehuddin
(Member)



Dr. Shamsad Ahmad
(Member)



Dr. Salah U. Al-Dulaijan
Department Chairman



Dr. Salam A. Zummo
Dean of Graduate Studies



27/11/17
Date

DEDICATED TO
MY BELOVED PARENTS AND MY LOVELY WIFE

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ABSTRACT

NAME SAMI SALEM HAMEED
TITLE DEVELOPMENT OF ALKALI-ACTIVATED BINDERS
UTILIZING INDIGENOUS INDUSTRIAL WASTE
MATERIALS
Major Field CIVIL ENGINEERING
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Due to environmental restrictions, there is a tendency worldwide to replace Portland cement with more environmental-friendly binders for use in building construction. Recently, research has been dedicated to the development of alkali-activated binders. These types of binders are produced by the activation of Si- and Al-rich materials utilizing alkaline solutions. Generally, fly ash is utilized as a source of Si and Al. However, other materials that may contribute these elements can also be utilized to develop alkali-activated binders. Since fly ash and silica fume are not available in the Kingdom of Saudi Arabia, other alternatives of producing alkali-activated binders need to be studied.

The aim of this study was to develop alkali-activated binders utilizing indigenous industrial waste materials, such as cement kiln dust (CKD), limestone powder (LSP), and bag house dust (BHD).

Compressive strength (f'_c) was measured as high as 25 MPa for alkali-activated CKD, which means that it can be used as a binding material that can replace Portland cement and result in a sound concrete mix with reasonable properties. The CKD activated alkali binder was developed with optimum ($\text{Na}_2\text{SiO}_3/\text{NaOH}$) ratio of 2.5. The maximum compressive strength of LSP and BHD alkali-activated binders was as low as 6.7 MPa and 9.0 MPa, respectively.

While corrosion measurements (according to ASTM C876) may not be valid for alkali activated binders, the alkali-activated binders exhibited very good sulfate resistance as indicated by very low loss of weight ($< 1.2\%$) after 270 days of exposure to ($\text{NaSO}_4 + \text{MgSO}_4$) solution.

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ABSTRACT (ARABIC)

الاسم سامي سالم حميد
عنوان الرسالة تطوير روابط منشطة بواسطة قلويات باستخدام مخلفات صناعية محلية
التخصص الهندسة المدنية
سنة التخرج ديسمبر 2017

بسبب القيود البيئية، هناك ميل في جميع أنحاء العالم لاستبدال الأسمنت البورتلاندي بروابط تكون صديقة للبيئة أكثر لاستخدامها في إنشاء المباني. في الآونة الأخيرة، تم تخصيص البحوث لتطوير روابط منشطة بواسطة قلويات. يتم إنتاج هذه الأنواع من الروابط من خلال تفاعل مواد غنية بالسيليكون والالمنيوم باستخدام محاليل قلوية. عموماً، يستخدم الرماد المتطاير (Fly Ash) كمصدر للسيليكون والالمنيوم. ومع ذلك، من الممكن استخدام مواد أخرى والتي يمكن أن تسهم هذه العناصر لتطوير روابط منشطة بواسطة قلويات. وبما أن الرماد المتطاير ودخان السيليكا (Silica Fume) غير متوفرين في المملكة العربية السعودية، فإن هناك حاجة لدراسة بدائل أخرى لإنتاج روابط منشطة بواسطة قلويات.

تهدف هذه الدراسة إلى تطوير روابط منشطة بواسطة قلويات باستخدام مخلفات صناعية محلية، مثل غبار قمارن الأسمنت (CKD)، ومسحوق الحجر الجيري (LSP)، وغبار الأكياس المنزلية (BHD).

تم قياس قوة الانضغاط (f_c) بـ (25) ميغا باسكال باستخدام (CKD) مما يعني أنه ثبت أنه يمكنها لاستبدال الأسمنت البورتلاندي لإنتاج مزيج خرساني مع خصائص معقولة. وقد تحقق ذلك مع النسبة المثلى من 2.5 من $(Na_2SiO_3/NaOH)$. ومع ذلك، لم يتم التعمق في دراسة كل من (LSP) و (BHD) أبعد من مرحلة الخلطات التجريبية (الحد الأقصى لمقاومة الانضغاط التي تحققت من قبل الخلطات التجريبية التي تم تحضيرها باستخدام (LSP) و (BHD) كانت منخفضة إلى حد 6.7 ميغا باسكال و 9.0 ميغا باسكال على التوالي وهي قيم منخفضة للغاية).

في حين أن قياسات التآكل وفقاً لمعيار ASTM (C876-08) قد لا تكون صالحة لهذا النوع من الأسمنت، قد أبدى هذا النوع من الخرسانة مقاومة جيدة جداً للكبريتات فقدان منخفض جداً من الوزن (أقل من 1.2٪) بعد 270 يوماً من التعرض لمحلول $(Na_2SO_4 + MgSO_4)$.

ماجستير في العلوم

جامعة الملك فهد للبترول والمعادن

الظهران، المملكة العربية السعودية

CHAPTER 1

INTRODUCTION

1.1: GENERAL

The main binding material in concrete is Portland Cement (PC). Up to this moment, no concrete is practically produced without using Portland cement. Other uses of Portland cement include: concrete mortars, stuccos and grouts for all types of building and construction.

Portland cement becomes this famous and widely spread and used as it provides advantages that no other building material provides. These advantages include: low cost, abundant availability of the constituent materials, easy to use and easy to implement. However, there is a major issue regarding the CO₂ footprint of cement.

One of the major problems associated with Portland cement manufacturing is the negative environmental impact that it causes by emitting a huge amount of CO₂. It is reported that one ton of cement produces almost 900 kg of CO₂. Only this year, 1.75 billion tons of CO₂ was emitted during the production of cement. Another issue is the use of significant volume of energy used for the production of cement.

Consequently, the new trend is towards the development of alternative binders for use in the building industry. If success is its ally, this will lead to a significant reduction in the

negative impact of concrete on the environment in a cost-effective way. With no additional charge (actually, it may be even less expensive than conventional concrete made with Portland cement), it makes it very attractive to be implemented in the building construction. Moreover, it is a breakthrough in concrete research to save the environment with a faster and easier than ever abiding with the internationally set limits of CO₂ emissions. Alkali-activated binders are being researched in countries around the world. Fly ash and other supplementary cementing materials are used as the primary constituent in the alkali-activated binders. However, there is a need to develop these binders utilizing other industrial waste materials. This study is a step in this direction.

1.2: RESEARCH SIGNIFICANCE

Many previous attempts towards an environmental friendly concrete production and usage included only partial replacement of Portland cement, typically 10-50 %, with a wide range of other supplementary cementing materials, typically waste materials [1].

A better route toward making concrete as a major construction material more environmental friendly is to study how the ingredients of conventional concrete can be revised. There are several important issues that need to be studied and well understood when doing so. There are two major points regarding the concrete ingredients:

- Less cement in concrete will have good impact on decreasing CO₂ emissions; cement replacement is a very good area of research.
- Less water in concrete design will have good impact on natural resources, water is more consciously utilized.

So, working on these two points might result in more green concrete mixtures when it comes to environmental impact.

This study utilizes the benefits of using alkali activators in the reduction of environmental impacts and moving toward more green development because it uses basic and natural resources in a very optimal way. Moreover, it involves the utilization of “local” natural resources and industrial waste materials to develop a more environmental friendly binder for concrete.

1.3: RESEARCH OBJECTIVE

The overall objective of this study was to develop alkali-activated binders utilizing indigenous industrial waste materials. The specific objectives were the following:

- (i) Select the optimum mix design for the selected materials,
- (ii) Evaluate the mechanical properties,
- (iii) Assess the pore structure and morphology of the developed green binders, and
- (iv) Investigate some of its durability characteristics.

1.4: RESEARCH OUTCOMES

The main outcomes of the reported research were the mixture composition and curing regime for the alkali-activated binders developed utilizing indigenous industrial waste materials.

Other outcomes include:

- ❖ Producing a binder free-of-cement, hence producing more green binder than Portland cement to help better save our environment.
- ❖ Utilizing local industrial by-products, which are already a waste, as a replacement material besides Portland cement removal (double environmental saving effort).
- ❖ Lowering carbon dioxide emissions up to 90% compared to conventional cement due to:
 - Decrease in the quantity of amount of calcium-based raw materials
 - Reduced manufacturing temperature
 - Decrease in the volume of fuel.
- ❖ Saving water since geopolymer concretes require less amount of water in their production
- ❖ Reducing the cost of the main binding material in concrete with this cost-effective new binders because:
 - Charges for the disposal of waste are avoided
 - Less cement is required to achieve similar strength as conventional binders
 - Less water is required in the new mixes
- ❖ Producing concrete with extra built-in features without any additional admixture or additive. Some of these features include:
 - Producing more durable concrete with regards to sulfate attack since the content of both C_3A and C_3S is reduced by the replacement of Portland cement.

- Producing concrete with built-in colors other than grey. Most likely, beige and brown which is different from the normal color of Portland cement.

1.5: RESEARCH PROGRAM

The reported research was conducted to develop a sustainable and environment-friendly binder. Three industrial waste materials were investigated to achieve the objectives of the reported research. The investigated binders are:

- Cement Kiln Dust (CKD)
- Limestone Powder (LSP)
- Bag House Dust (BHD)

Figure 1.1 shows the main activities involved in the research program. The details of this methodology can be broken down as follows:

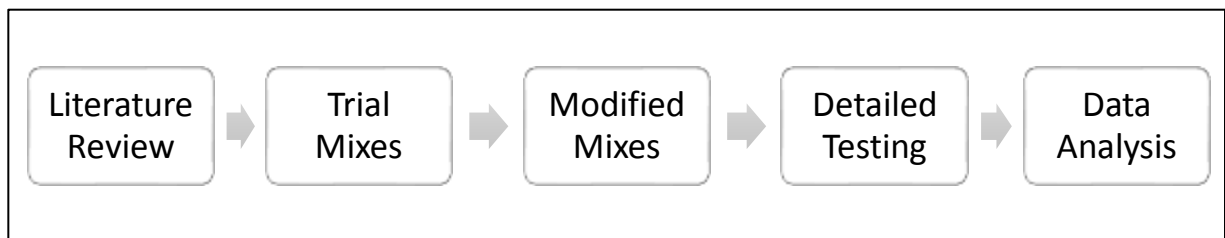


Figure 1.1: Main Activities of the Research Program

1.5.1: TASK I: LITERATURE REVIEW

An in-depth literature review was carried out in order to investigate more in the area of the alkali-activated binders. Moreover, more explorations were done on how these materials can be utilized to the best of our ability to develop a sustainable alkali-activated binder.

1.5.2: TASK II: EXPERIMENTAL STAGE-1: TRIAL MIXES

Initially, this stage aimed to investigate the possibility of developing a binder utilizing the proposed industrial waste materials (CKD, LSP, and BHD). The main property to look at was the compressive strength as a first indication for the proven capability of the trial mixtures.

1.5.3: TASK III: EXPERIMENTAL STAGE-2: MODIFIED MIXES

More revised trial mixes were prepared in this task. Some parameters that were investigated included:

- Curing temperature and time
- Alkalinity

1.5.4: TASK IV: EXPERIMENTAL STAGE-3: DETAILED TESTING

Based on the trial mixtures (Task II and III), optimum mixture composition for each of the industrial waste material was selected. Specimens of varying geometry were prepared according to standard practice tests and they were tested for:

- Compressive strength
- Flexure strength
- Modulus of elasticity
- Scanning Electron Microscopy (SEM)
- Energy Dispersive Spectroscopy (EDS)
- Chemical Compositions

- Reinforcement Corrosion
- Sulfate Attack

1.5.5: TASK V: DATA ANALYSIS

The data obtained from the experimental program were utilized to:

- 1- Investigate the parameters affecting the properties of the developed green binders
- 2- Evaluate the mechanical properties and durability characteristics of the developed green binders,
- 3- Assess the pore structure and morphology of the developed green binders, and
- 4- Assess strength and service-life of the developed alkali-activated binders.

CHAPTER 2

LITERATURE REVIEW

2.1: CONVENTIONAL CONCRETE BINDER (PORTLAND CEMENT)

Conventionally, concrete is produced by mixing various ingredients. There are basic (main) ingredients, without their existence in the mix it will not be concrete, and there are other secondary ones. Basic ingredients are water, aggregate, sand, and cement while chemical and mineral admixtures are some secondary ingredients added to concrete mix in order to achieve certain properties to produce concrete. Concrete production is time-sensitive. Once the ingredients are mixed, workers must put the concrete in place before it hardens.

Without the need for heat or other external processing agents, a fluid suspension is transformed into a rigid solid at room temperature and with minimal bulk volume change; this is what makes concrete mix almost a magical thing. Conventional concrete is a mixture of Portland cement, water, aggregates, and admixtures, chemical and otherwise. Since Portland cement is the main binding material in concrete, it has to grab special attention from people who are working with concrete, as designers, researchers, etc. [1].

When initially mixed, Portland cement and water rapidly form a gel of tangled chains of interlocking crystals, and components of the gel continue to react over time. Initially the gel is fluid, which improves workability and aids in placement of the material, but as the concrete sets, the chains of crystals join into a rigid structure, counteracting the fluidity of the gel and fixing the particles of aggregate in place. During curing, the cement continues

to react with the residual water in a process of hydration. In properly formulated concrete, once this curing process has terminated the product has the desired physical and chemical properties. Among the qualities typically desired, are mechanical strength, low moisture permeability, and chemical and volumetric stability [2].

Thorough mixing is essential for the production of uniform, high-quality concrete. For this reason, equipment and methods should be capable of effectively mixing concrete materials containing the largest specified aggregate to produce uniform mixtures of the lowest slump practical for the work [2].

In this study, one of the basic mix ingredients was tried to be replaced, i.e. Portland cement. It was replaced by materials that is commonly used as additives but they were tried and used as the main binding cementitious materials in our concrete. These are CKD, LSP, and PHD. Their properties, technical and other information will be presented and discussed in details later in this report.

2.2: QUALITATIVE AND QUANTITATIVE BINDING MECHANISM OF PORTLAND CEMENT

Globally, the quantitative understanding of the underlying mechanism of hydration process is advancing rapidly. It obeys well-established principles of chemical thermodynamics and kinetics. However, there are many things that are still not very clear. For instance, the reasons for the slowdown in the reaction after about 10 hours are still not clear. In addition, the on-going hydration mechanisms beyond the first day are not yet well understood and clarified, although they are very important for long-term properties of concrete. Concrete

requires very little cement paste to glue the aggregates together for better strength, thus avoiding volume stability problems caused by large amounts of paste. Therefore, the higher content of paste in pores of concrete will dilute the bond between aggregate, then the strength of concrete will be decreased, but practically, more cement content will help to achieve better compressive strength and other properties. Therefore, more in-depth understanding of the kinetics of cement hydration is really required [3].

As illustrated in Figure 2.1, limestone and clay are the major components of Portland cement with principal constituents being CaO and SiO_2 which form the hydraulic calcium silicates, the active compounds in cement, when subjected to temperature as high as 1450°C . In Portland cement manufacturing, generally 25% of the total mix is liquid and hence the process is neither a complete fusion nor a pure solid-state sintering; it is called clinkering. Portland cement clinker can be described via its four major oxides (CaO - SiO_2 - Al_2O_3 - Fe_2O_3). When the clinker cools to a sub-solidus temperature, the four major compounds; C_3S , C_2S , C_3A , and C_4AF are formed. Later on, gypsum is added during grinding process for delaying the setting of cement [4].



Figure 2.1: Simplified Portland cement Manufacturing Process

Besides the major compounds in Portland cement, there are other important minor ones. These include: alkali metals, alkali metal oxides, and many others. The first one increases the Na_2O content which modifies polymorphism of C_3A . Also, it decreases C_2S and increases C_3S with the possible occurrence of free lime. The second one, increases the viscosity and decreases the surface tension of the liquid phase. Actually, the analysis of minor elements in cement can be used to identify the origin of cement, to explain the reasons of the deterioration of the structure, and to identify Mg, Sr, and Mn as “fingerprints” [5].

Setting and hardening of concrete is very important to be well understood and clearly identified. It is the result of complex chemical reactions between cement and water (Hydration process) in the cement paste binder. It is worth mentioning that silicates are responsible for the strength of cement pastes but in the first seven days the alite content controls the development of strength. With regard to porosity, the higher the volume of calcium silicate hydrate (C-S-H) in the microstructure the less the overall porosity because (C-S-H) has a lower specific gravity than CaOH_2 ; thus creating a durable concrete [6].

Clearly, the more the knowledge and technology advancements that will happen in the area of understanding the reactive phases of the hydration mechanisms and process details the better and easier the involvement of supplementary cementitious materials. Even better, the development of new clinker will be much easier and trails will be minimized to find new more sustainable binders for future concrete.

2.3: ENVIRONMENTAL IMPACT OF CURRENTLY USED BINDERS

Waste materials are non-dispensable in most cases and thus, they remain unused for hundreds and thousands of years. Since they are non-biodegradable, an environmental crisis is caused by their negative impact all around the world. Another negative environmental impact is CO₂ emissions in which Portland cement is a major contributor.

CO₂ concentration level in the air is rising at an alarming rate. As shown in Figure 2.2, CO₂ concentration in the air was around 280 ppm in 1750s. By the end of World War-II, CO₂ levels had risen to 310 ppm. Then, the figure jumped to 365 ppm by the year 2000 and currently, the CO₂ concentration made a big jump reaching to about 393 ppm.

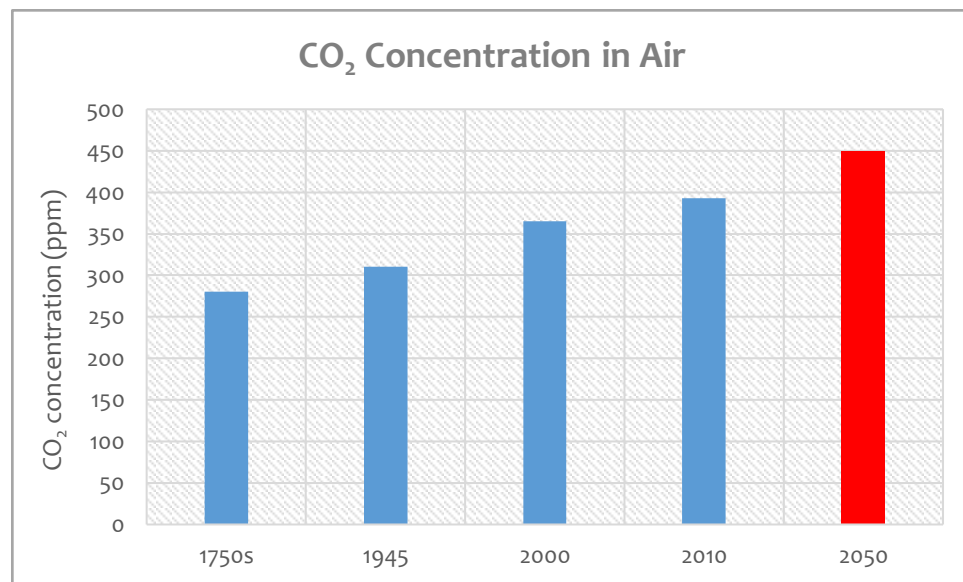


Figure 2.2: CO₂ Concentration Levels with Time

So, it is very clear that the concentration of CO₂ is increasing with time, which means this problem is accelerating in a scary scale [7-8].

With the very large annual worldwide Portland cement production (approaching 3 Gt), cement alone contributes about 5% of CO₂ emissions on the planet. In this year, cement industry contributed about 1.74 billion ton (= 1,740,000,000 ton) of CO₂ compared to 1.7 billion ton in 2009. In cementitious materials, nearly all sources of lime (CaO) comes from limestone CaCO₃ directly or indirectly. So, lime content has a direct bearing on the associated emission on CO₂. It is worth mentioning that the average CO₂ emission associated ONLY with the grinding process is about 0.1 ton of CO₂ per ton of cement [9].

2.4: PREVIOUS EFFORTS TO SAVE ENVIRONMENT AND REDUCE THE IMPACT OF CURRENTLY USED BINDERS

There are two main ways that is well known to reduce the CO₂ footprint of concrete without reducing its usefulness as a construction material. One way is the use of more efficient concrete regarding CO₂ emissions. In other words, use alternative fuel with less CO₂ footprint. The other way is to reduce the average CO₂ footprint of hydraulic binders by increased replacement of Portland cement by the supplementary cementitious materials [10]. Other possibility is the development of binders that do not use the clinkering process.

With the exception of limestone, the supplementary cementitious materials are silico-aluminate materials with lower calcium content than Portland cement, extending along silica to alumina ratio of about “two”. The durability of limestone mortar is mainly due to fillers, such as alumino-silicates [3].

Even if the two ways mentioned earlier are followed and pushed to their maximum practical limits, the global cement industry will only be about half-way to meet the limit set by the International Energy Agency (IEA). IEA is targeting to limit the CO₂ emissions to a level

of 450 ppm in 2050 as shown in Figure 2.2 earlier and according to their proposed “Blue Map” scenario which is shown in Figure 2.3 [11].

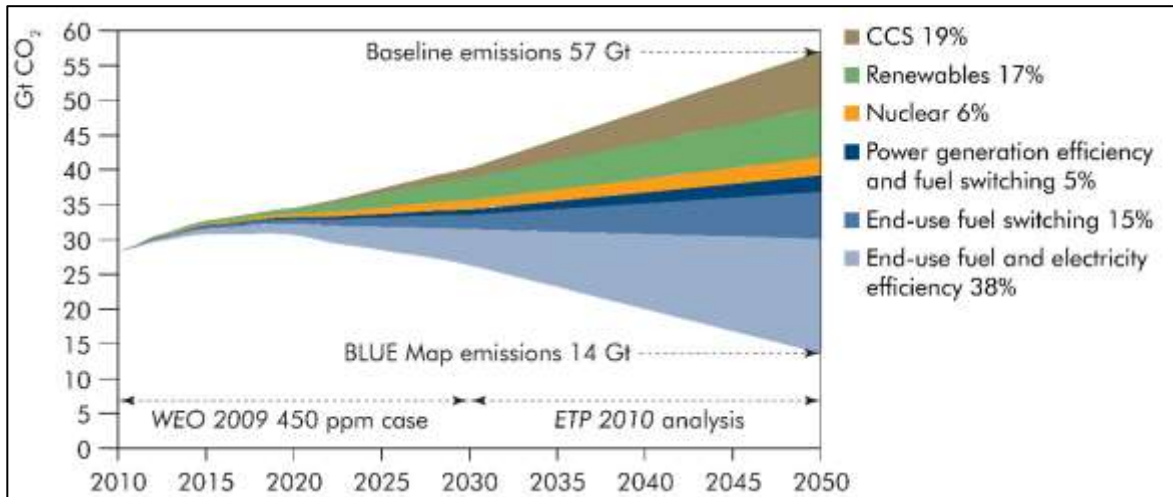


Figure 2.3: Blue Map Scenario [11]

This is because the IEA’s analysis assumes that CO₂ that is captured and sequestered (CCS) will have to be applied to the cement industry. This is very difficult and not practical due to many reasons. Most importantly, it is a very expensive process by itself which means the cost of cement production is going to increase leading to more expensive concrete with same properties like normal concrete. Economically, it means to pay more for nothing. Also, it is unattractive to stock the excess CO₂ underground in pressurized reservoirs. Moreover, new facility is required to be installed as a part of cement manufacturing process which is making the manufacturing process little more complicated. So, cheaper and simpler way is needed to effectively solve this issue [10].

There is a need to think more comprehensively regarding saving the environment. In a very simple breakdown, this should start by defining all possible saving ways for investigation. Then, evaluation of how sustainable they are is to be carried out. Finally, evaluating their applicability, potentials, difficulties, advantages and disadvantages in order to clearly measure and reach the savings that are satisfactory and meeting the set limits by international organizations concerned with saving the environment.

Waste materials, particularly those produced during industrial production can be recycled to produce new binders with a view to protect the environment. Although, few studies have been conducted to develop alternative binders; there is much more to be done to achieve the objective of reducing the carbon footprint by the building industry [12].

Earlier, there was no major issue, especially political, regarding saving the environment but recently, the construction industry is under increasing pressure to reduce its impact on the environment with the increased global attention and awareness towards more sustainable and more environmental friendly orientations [3].

Due to its very low cost and nearly zero associated CO₂ emission, limestone powder is a particularly interesting supplementary cementitious material. It is clear that this can react with calcium aluminate to give space filling calcium carbo-aluminate. Four promising alternative binders to Portland cement are available which are:

- calcium aluminate cement
- calcium sulfoaluminate cement
- alkali-activated binder
- supersulfated cements [1, 3].

2.5: ALKALI-ACTIVATED BINDERS

Alkali-activated binders are currently under investigation. They are new generation of binders. The alkali-activation (often called “geopolymerization”) is a method by which the low reactivity of many pozzolanic materials may to some extent be compensated by activation with concentrated basic alkali metal solutions. They are alumino-silicate binders, i.e. developed using alumina-silica material [7].

Geopolymer cements are made from mixing of water-soluble alkali metal silicates (e.g. sodium silicate) and aluminosilicate mineral powder (e.g. fly ash). In case of full replacement, it is useful to understand how the new replacing materials convert to cement. Although binders from low calcium aluminosilicate (clay) or calcium-free aluminosilicate (clay) and alkaline metal solution were understood first in 1967, a significant technological growth in research related to this area only has been seen during the last 12 years. With the most burning challenge facing the world today, it is very critical to understand the underlying mechanisms in order to progress and continually lower environment impact, doing more with less [7].

Geopolymer materials are inorganic polymers based on alumina and silica units; in a highly alkaline environment, they are synthesized from a wide range of dehydroxylated alumina-silicate powders mixed with alkaline silicate. Geo-polymeric materials can be produced from a wide range of alumina-silica, including natural products and by-products. The word “GEOPOLYMER” is used to highlight:

- The presence of natural mineral analogues in their hydration products (GEO)

- The similarities in the mechanism governing the formation of these binders and natural geological materials (GEO)
- Their polymeric structure (POLYMER) [5].

The main benefit of geopolymer cement is the reduction in environmental impacts and moving toward a more sustainable development because it uses basic and natural resources in a very optimal way. These binders are less energy consuming and results in lower CO₂ emissions [12].

The setting time and final strength are affected by the calcium content in cement and there are indications that it also does the same for geopolymer concrete properties. The alkali content of the reacting minerals could have a significant effect on strength development of geopolymers since in geopolymerization, the dissolution reaction and polycondensation steps involve alkali metals.

Alkali-activated binders do NOT react on their own because of no alkalinity. So, alkaline material is required to accelerate their reactions. More specifically, the alkali-activation is a method by which the low reactivity of many pozzolanic materials may to some extent be activated with alkali material solution. Alkali-activated binders are based on alumina and silica units in a highly alkaline environment which results in forming an alkali aluminosilicate binder gels (See Figure 2.4).

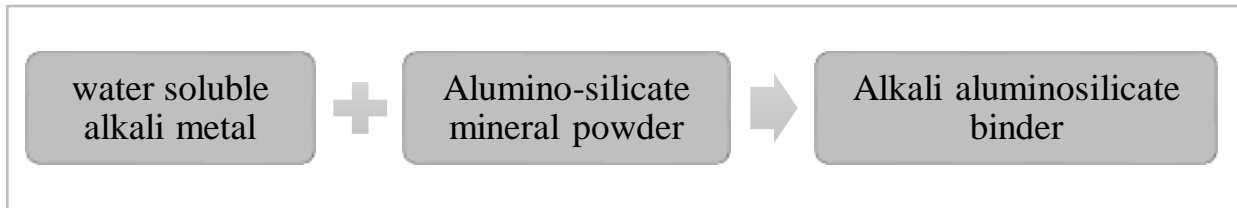


Figure 2.4: Alkali-activation process

Mostly, these types of binders need thermal treatment, i.e. curing to be done in a temperature higher than room temperature to be activated.

The chemical process and the consumption of significantly less fuel reduces the carbon dioxide emission for geopolymeric cement production because geopolymer cements neither rely on the calcinations of calcium carbonate nor require high-temperature kiln-temperature with large expenditure of fuel [1].

The variability of local materials is a major challenge. So, it is very essential for this idea of Portland cement replacement to address this issue clearly and with practical solutions. Using wide range of different cementitious combination might help a lot in this regard [3].

There are many advantages of using Alkali-activated binders in producing concrete mixes. Below are some of these:

- Utilizing alkali-activated binder will make concrete more green because of:
 - Reduction of CO₂ emissions that results from PC manufacturing
 - Saving required energy to manufacture PC
 - Saving water since alkali-activated concrete is expected to:
 - Have less water in their mix

- Does NOT need water curing
- Reducing cost of binder because:
 - No PC is required → saving the cost of PC
 - Disposal costs of industrial waste materials are avoided
 - Less water is required → saving the cost of water
- Using alternative binder that is available locally

Some materials were previously investigated in this area. This includes:

- Fly ash
- Silica fume
- GGBFS
- Natural Pozzolan.

While some other potential materials were not previously investigated in this area including:

- Cement Kiln Dust (CKD)
- Limestone Powder (LSP)
- Bag House Dust (BHD)
- Oil Ash (OA)

CHAPTER 3

METHODOLOGY OF RESEARCH

3.1: USED BINDERS

Three industrial waste materials (i.e. CKD, LSP, and BHD) were selected to be tested as Alkali-activated binders. In this section, an overview of the chemical compositions as well as other characteristics of these materials are discussed.

3.1.1: Cement Kiln Dust (CKD)

Cement Kiln Dust (CKD) is a by-product of cement clinker. It is generated during the dry process of manufacturing the cement clinker in large quantities. It is found in a form of very fine powder which makes it very hard to handle and dispose. In 2010, around 3.4 million tons of CKD was resulted from this manufacturing process. It is estimated that every year, 6 to 7 % of CKD is resulted from Portland cement clinker during the manufacturing process [13-14].

Table 3.1 shows the chemical compositions of CKD.

Table 3.1: Chemical Composition of CKD

Constituent	Weight %
CaO	49.3
SiO ₂	17.1
Al ₂ O ₃	4.24
Fe ₂ O ₃	2.89
K ₂ O	2.18
MgO	1.14
Na ₂ O	3.84
P ₂ O ₅	0.12
Equivalent alkalis (Na ₂ O + 0.658K ₂ O)	5.27
SO ₃	3.56
Chloride	6.90
Loss on ignition	15.8
BaO (µg/g (ppm))	78.2
Cr ₂ O ₃	0.011
CuO	0.029
NiO	0.012
TiO ₂	0.34
V ₂ O ₅	0.013
ZnO (µg/g (ppm))	65.8
ZrO ₂	0.011

CKD is originally considered a waste by-product of cement clinker. However, there are some uses and applications that in which CKD can be useful and thus, avoiding disposal and hence environmental pollution. A major part of this study is about the use of CKD as a cementitious material in concrete manufacturing replacing cement partially and/or totally.

Many studies incorporated the use of CKD in concrete, i.e. as cementitious material. Al-Harthy et al. [15] concluded that the use of CKD with certain partial replacement of Portland cement to produce concrete and mortar does not show any negative effect.

Maslehuddin et al. [16] concluded that the use of up to 5% CKD as a partial replacement of Portland cement in producing concrete does not show a significance difference in compressive strength and drying shrinkage compared with concrete produced without CKD.

3.1.2: Limestone Powder (LSP)

Crushing carbonate rocks is primarily done to produce coarse aggregate in central and eastern regions of K.S.A. A fine powder results from this process, which is the so called “Limestone Powder”. This by-product is commonly used as a filler material in producing concrete all over the world which helps in enhancing the properties of concrete technically, economically, environmentally and/or otherwise.

Achieving strength earlier as well as controlling bleeding are some of the technical issues that concrete mixes that contains a specific amount of LSP help to improve. Economically, such type of concrete mixes are costing less than conventional concrete mixes which they do not contain LSP. By using less cement, this means producing less CO₂ that is being emitted in the air and cause environmental pollutions.

Table 3.2 shows the chemical compositions of LSP.

Table 3.2: Chemical Composition of LSP

Constituent	Weight %
SiO ₂	11.79
CaO	45.7
Al ₂ O ₃	2.17
FeO ₃	0.68
MgO	1.80
K ₂ O	0.84
Na ₂ O	1.72
Na ₂ O + (0.658K ₂ O)	2.27
Loss on Ignition	35.10
Moisture	0.20

Many studies investigated the effects of LSP use in preparing cement and concrete mixes.

Liu and Yan [17] showed that LSP filling effect can make the paste matrix and interfacial transition zone between matrix and aggregate denser, which improves the performance of concrete despite the fact that it does not possess pozzolanic properties.

Heikal et al. [18] reported that LSP addition in pozzolanic cement helps to reduce the total porosity. In addition to that, it reduces the initial and final setting time. Moreover, lower diffusion coefficient of chloride ions is noted and lower heat of hydration and higher compressive strength as well.

3.1.3: Bag House Dust (BHD)

Bag House Dust (BHD) is a by-product of steel making process. It is generated during the electric arc furnace steel making. It is a very fine powder which harmfully forming most of the smoke and fume that is coming out of the furnace during the steel production. The “Bag House Dust” name comes from the way that this product is being drawn through

cooling pipes in specially designed bag filters. Each ton of steel produced, generates 15 to 20 kilograms of BHD. This means around 8,242 tons of BHD is annually generated in Saudi Arabia since the reported annual steel production is around 471,000 tons [19-20].

BHD is a source of a major environmental problem due to the fine dust nature of this product which is released to the atmosphere causing pollution. Also, it is considered as a hazardous material and therefore it must be disposed or collected in a special way.

Table 3.3 shows the elemental compositions of BHD.

Table 3.3: Elemental Composition of BHD

Constituent	Weight %
Aluminum	0.7
Calcium	9.39
Cadmium	0.0004
Copper	0.06
Iron	33.6
Potassium	1.7
Magnesium	2.3
Manganese	1.8
Sodium	2.6
Nickel	0.01
Lead	1.31
Phosphorous	0.13
Silicon	2.38
Tin	0.03
Sulphur	0.57
Titanium	0.09
Zinc	10

BHD has a retarding effect on the setting time. Zinc and pozzolanic materials that this by-product contains makes it a good candidate in preparing concrete mixes with enhanced properties, especially compressive and shearing strength as well as abrasion.

Many studies show that BHD is a very good substitute material in concrete preparation to achieve better mixes. Maslehuddin et al. [21] reported that 2% replacement of BHD in both OPC concrete as well as blended cement concrete helps to enhance many properties. Setting time and slump retention is getting higher values with 2% replacement with BHD. In addition to that, there was a reduction in water absorption, a lower chloride permeability, and higher corrosion resistance. Also, there was a gain in strength with concrete that has the BHD replacement compared to those concretes in which there were no BHD replacement.

De souza et al. [19] found that BHD helps to achieve higher compressive strength (10 to 20 percent addition of BHD). On top of that, chloride penetration decreased and setting time increased with the help of BHD.

3.2: TRIAL MIXES

This stage aimed to primarily investigate the possibility of using three materials, i.e. CKD, LSP, and BHD, as a binder by full replacement of ordinary Portland cement in preparing concrete mixes. The main property to look at in this stage was the compressive strength as a first indication of the proven capability of the trial mixtures of these materials under investigation.

Initially (150 kg/m³) of “sodium silicate solution” was assumed. Then by changing this proportion, the effect of sodium silicate content on mix properties was investigated (basically compressive strength in this stage). By the help of current studies at KFUPM Research Institute on similar research topics, the mix contents for the trial mixes were determined and the parameters were subsequently changed to find a working mix and see the effect of these parameters on concrete properties.

Figure 3.1 indicates the ingredients used in the preparation of alkali-activated concrete.

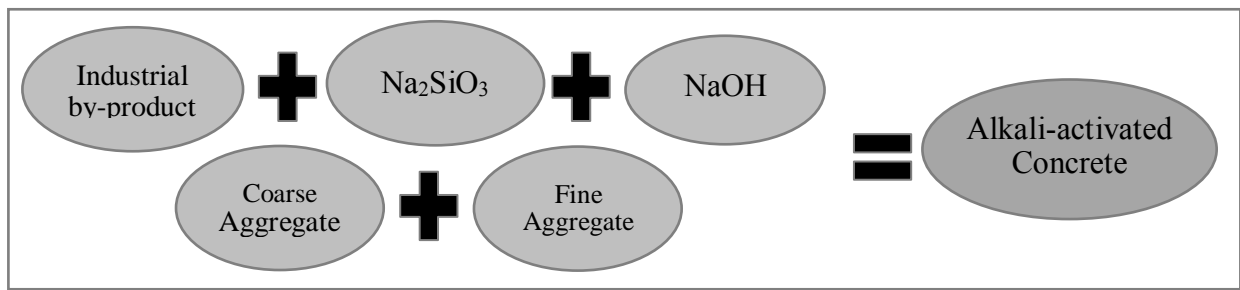


Figure 3.1: Alkali-activated concrete Major ingredients

The details of the materials used in this study are described in the following sub-sections.

3.2.1: Concrete Mix Ingredients

❖ Coarse Aggregates:

Aggregates are the raw materials that are an essential ingredient in concrete. They need to be clean, hard, strong particles free of absorbed chemicals or coatings of clay and other bad materials that could cause the deterioration of concrete.

The used coarse aggregates were crushed limestone from local quarries. The specific gravity and absorption of the used coarse aggregates were 2.6 and 2.4 % respectively,

which were determined in accordance of ASTM C127 [22]. The used coarse aggregates size combination in the tested mixes is:

- 90% of size No.4 (4.75 mm)
- 10% of size No.8 (2.36 mm)

❖ **Fine Aggregate:**

Dune sand of which Saudi Arabia has an abundant quantities was used as a fine aggregate.

The average values of specific gravity and absorption were 2.65 and 0.4 % respectively.

Table 3.4 shows the grading of the used dune sand.

Table 3.4: Grading of the fine aggregate used in the study

ASTM Sieve No.	Size (mm)	Percentage Passing (%)
4	4.75	100
8	2.36	100
16	1.18	100
30	0.6	75
50	0.3	10
100	0.15	5

❖ **Base Materials:**

In this study, CKD, LSP, and BHD were used as base materials in preparing the alkali-activated concrete. The chemical compositions and other information for these alternative cementitious materials were presented earlier in this report. Portland cement (PC) was not used in the preparation of any of the concrete mixes in which were trying to prepare and investigate. PC was totally replaced with these materials which were being tested as the

main binders in the developed alkali-activated concrete. Figure 3.2 through 3.4 show the samples of the investigated materials.



Figure 3.2: Cement Kiln Dust (CKD) Sample



Figure 3.3: Limestone Powder (LSP) Sample



Figure 3.4: Baghouse Dust (BHD) Sample

❖ **Water (H₂O):**

Water was not added separately to the mix. The only water used was the water that is contained in the solutions used to prepare 16 molar sodium hydroxide solution and sodium silicate solution.

❖ **Sodium Hydroxide (NaOH)**



Figure 3.5: Sodium Hydroxide Pellet

Sodium Hydroxide is a white solid and it is an alkali salt which is available in the form of pellets, flakes, granules, and as solutions in a number of different concentrations. It is used to activate the sodium silicate to work as the main cementitious material. A 16 molar (mol/L) sodium hydroxide solution was prepared in the laboratory using distilled water and sodium hydroxide pellets according to the following equations:

Atomic Weight of (NaOH) = 40 g

1 molar solution will have 40 grams of NaOH per Liter of water

16 molar solution will have 640 grams of NaOH per Liter of water

So, the following formula is used to prepare 100 mL of 16 molar sodium hydroxide solution:

(NaOH) _{16M} = “100” ml of water + “64” grams of Sodium hydroxide pellets

❖ **Sodium Silicate (Na₂SiO₃)**



Figure 3.6: Sodium Silicate Solution

This was the main variable (in terms of percentage content in the prepared mixes) component in the trial mixes that was changed and the effect of its content on the mix properties as well as compressive strength was determined. It helps the base material (i.e.

CKD, LSP, and BHD) to work as the main binding material in concrete mixes since it is totally replacing the ordinary Portland cement in concrete mix in which this study is trying to prepare and test. As this study is going to show later, this is the main component that affects properties of both fresh and hardened concrete mixes. It affects the homogeneity, workability, compressive strength and many other concrete properties of the alkali-activated binders.

Sodium silicate solution which is being used in this study consists mainly from (35 – 40%) Sodium Silicate and (60 – 65%) water.

Sodium silicate has an essential role since it contains silica which works as a binding material in the new concrete under investigation.

For more information about physical properties, chemical properties, and many other useful information related to sodium silicate, please refer to the detailed data sheet in Appendix-A.

3.2.2: Mixing Procedure

In this stage, each of the three potential binders was tested separately to see if any can act as the main binding component in concrete by achieving an acceptable compressive strength. Then it was proceeded to the next stage with the mixes that show, at least, reasonable compressive strength. In the next section, experimental data, illustrations, other information and details of the three potential binders are presented and discussed in details.

A sample size of 50x50x50 mm cube was used in this stage, such small samples size was selected for many reasons. They save time in mixing. They are easy to handle and move. Also, it helps to save LAB resources because many mixes are going to be prepared since

this is a totally new concrete mix and it was expected that many trial mixes will have several problems and issues to investigate and look at to reach an acceptable concrete recipe.

In the following sub-sections, further mixing details of the selected three base materials (i.e. CKD, LSP, and BHD) will be presented. Also, the way that mixed contents and parameters are modified to see the effect of each one are going to be discussed for each base material.

3.2.3: Trial Mixes with CKD

CKD is selected as a potential cementitious material that is going to replace Portland cement in concrete mixes. It is considered as a potential candidate that might successfully replace Portland cement since the chemical compositions of it shows high amount of “SiO₂” as well as good amount of “CaO” and it is well known that mainly calcium silicates are the only contributors to conventional concrete strength.

To initially investigate the feasibility of using CKD as a potential binder and as an initial trial with the help of current studies at KFUPM Research Institute on similar research topics, the mix contents for the trial mixing stage were selected and the parameters were varied to find a working mix and see the effect of these parameters on concrete properties.

By assuming a certain amount of “sodium silicate solution” then changing the alkali to powder (A/P) Ratio. This was the first parameter to check its effect on the prepared mixes. Also, the sodium silicate to sodium hydroxide ratio was changed several times to investigate its effect on our mixes and its properties especially at mixing and casting times. After that, liquid to powder ratio was also looked at to see what kind of changes happens to the mixes if this ratio was varied. Moreover, other parameters including but not limited

to adding superplasticizer as well as extra water were investigated to see their effect, if any, on such new concrete type which will be shown later in this report

The mixing ingredients of the CKD mixes were as follows:

- CKD
- #4 coarse aggregate (4.75 mm)
- #8 coarse aggregate (2.36 mm)
- Fine aggregate
- Sodium hydroxide solution
- Sodium silicate solution

The quantities of each ingredient will be shown in details later in this report in the part that describes the prepared mixes and all the details of the related experiments and tests.

At this stage, the curing temperature was fixed to be (60° C) inside the oven for 24 hours. CKD content was fixed at 400 kg/m³. A 3:2 ratio of coarse to fine aggregates was fixed and used in all the experiments. A 9:1 ratio of #4 to #8 coarse aggregates was also fixed.

Regarding water content and as mentioned earlier, the only water used was the water that is already contained in the sodium silicate and sodium hydroxide solutions. Initially, no water was intended to be added as a separate ingredient in the mixes under investigation (except for certain research purposes in experimental trials).

❖ **Trial Mix# 1**

Mix details:

In this trial mix, the mixed quantity was intended to fill 9 cubic molds of (50 x 50 x 50 mm) size. The ingredients of this mix are shown in Table 3.5.

Table 3.5: Quantity of each ingredient of Mix# 1

Ingredient	Quantity (kg / m³)
#4 coarse aggregate	1019.7
#8 coarse aggregate	113.5
Fine aggregate	755
CKD	400
16 molar sodium hydroxide solution	57
Sodium silicate solution	150

No additional water was added to this mix. Neither superplasticizer nor stabilizer were added to the mix. With the above ingredients, the ratio of sodium silicate to sodium hydroxide was 2.63 and the alkali to cementitious material was 0.518.

Observations:

The mix was NOT homogeneous (the mixing ingredients don't get mixed well to form a homogeneous mix to fill the mold) and it was considered an unsuccessful trial.

❖ Trial Mixes (2 to 4)

Mixing details:

In these trial mixes, the alkalis/powder ratio was changed and the mixing was monitored to see the effect of that on achieving better concrete mix homogeneity. This ratio was changed to 0.78, 0.86, and 0.90 for mixes 2, 3, and 4 respectively. Table 3.6 summarizes mixing data for mixes (1 to 4).

Table 3.6: CKD Trials (1 to 4) Summary Data

Mix. No.	CKD content (kg/m ³)	Na ₂ SiO ₃ (kg/m ³)	water in Na ₂ SiO ₃ (Liter)	NaOH 16M (kg/m ³)	water in NaOH (Liter)	Water / Powder	Liquid / Powder	Alkali / Powder	Na ₂ SiO ₃ / NaOH
1	400	150	93.8	57	34.8	0.09	0.52	0.52	2.63
2	400	225	140.6	86	52.5	0.13	0.78	0.78	2.62
3	400	250	156.3	95	58	0.15	0.86	0.86	2.63
4	400	260.9	163.1	99.1	60.5	0.15	0.90	0.90	2.63

Observations:

These mixes were better than the previous one in terms of homogeneity. They were all homogeneous mixes and all ingredients were well mixed forming a good homogeneous mix.

Unfortunately, these mixes were NOT workable at all and got stuck in the mixing bowl very rapidly. Thus, increasing the alkalis/powder ratio helped in solving homogeneity problems but another problem appear in concrete workability. So, these mixes were considered unsuccessful trials.

❖ Trial Mixes (5 to 8)

Mixing details:

Trying to solve the workability problem that was faced in the previous mixes so far, the effect of adding superplasticizer to the mixes was investigated. “Sika ViscoCrete-10 R” is the superplasticizer that was used in these mixes. The data sheet of this superplasticizer is attached in Appendix-B. The ingredients of mixes 5, 6, 7, and 8 were the same as mix# 2 except that for the additional amount of superplasticizer of 3, 6, 9, 12 liters, respectively.

Table 3.7 summarizes mixing data for mixes (5 to 8).

Table 3.7: CKD Trials (5 to 8) Summary Data

Mix. No.	CKD content (kg/m ³)	Superplasticizer (Liter/m ³)	Na ₂ SiO ₃ (kg/m ³)	water in Na ₂ SiO ₃ (Liter)	NaOH 16M (kg/m ³)	water in NaOH (Liter)	Water / Powder	Liquid / Powder	Alkali / Powder	Na ₂ SiO ₃ / NaOH
5	400	3	225	140.6	86	52.5	0.13	0.79	0.78	2.62
6	400	6	225	140.6	86	52.5	0.13	0.79	0.78	2.62
7	400	9	225	140.6	86	52.5	0.13	0.80	0.78	2.62
8	400	12	225	140.6	86	52.5	0.13	0.81	0.78	2.62

Observations:

Unfortunately, these mix were still NOT workable at all and got stuck in the mixing bowl very rapidly. Thus, adding superplasticizer even with a very high dosage, even higher than the maximum recommended values by the manufacturer, did not show any improvement

in making the mix more workable and these mixes were being considered unsuccessful trials.

So, another parameter was needed to be investigated to improve the mix workability in the coming trials.

❖ Trial Mixes (9 to 12)

Mixing details:

As another way of trying to enhance mix workability, adding additional water to the mix was tried in coming four mixes. Normally, adding water to conventional concrete mixes results in a more workable mixes but also it dramatically reduces strength of concrete. That is why the choice of adding water to the mix was not the best choice and often not jumped to as a solution to workability.

Additional separate volume of water of 5, 10, 15, and 20 liters was added to mixes 9, 10, 11, and 12 respectively. Table 3.8 summarizes mixing data for these mixes.

Table 3.8: CKD Trials (9 to 12) Summary Data

Mix. No.	CKD content (kg/m ³)	Water (Liter/m ³)	Na ₂ SiO ₃ (kg/m ³)	water in Na ₂ SiO ₃ (Liter)	NaOH 16M (kg/m ³)	water in NaOH (Liter)	Water / Powder	Liquid / Powder	Alkali / Powder	Na ₂ SiO ₃ / NaOH
9	400	5	225	140.6	86	52.5	0.14	0.79	0.78	2.62
10	400	10	225	140.6	86	52.5	0.16	0.80	0.78	2.62
11	400	15	225	140.6	86	52.5	0.17	0.82	0.78	2.62
12	400	20	225	140.6	86	52.5	0.18	0.83	0.78	2.62

Observations:

Although too little improvement was observed each time the water content was increased for these mixes, still these mixes were NOT good enough when it comes to workability and got rapidly stuck in the mixing bowl. Thus, inadequate workability was achieved with even 20 liters of water added in mix# 12. So, these trials were considered unsuccessful trials.

❖ Trial Mixes (13 to 16)

Mixing details:

In these mixes, sodium silicate to sodium hydroxide (Na_2SiO_3 / NaOH) ratio was examined checking how it was affecting the prepared mixes. The ratio was decreased to 2.00 and used for all of these four mixes while the ratio of alkalis/powder was varying i.e. 0.52, 0.70, 0.79, and 0.90 for mixes 13, 14, 15, and 16 respectively. See Table 3.9 for mixing data of mixes (13 to 16).

Table 3.9: CKD Trials (13 to 16) Summary Data

Mix. No.	CKD content (kg/m ³)	Na_2SiO_3 (kg/m ³)	water in Na_2SiO_3 (Liter)	NaOH 16M (kg/m ³)	water in NaOH (Liter)	Water / Powder	Liquid / Powder	Alkali / Powder	Na_2SiO_3 / NaOH
13	400	138.1	86.3	69.1	42.2	0.11	0.52	0.52	2.00
14	400	186.6	116.6	93.3	56.9	0.14	0.70	0.70	2.00
15	400	211	131.9	105.4	64.3	0.16	0.79	0.79	2.00
16	400	240	150.0	120	73.2	0.18	0.90	0.90	2.00

Observations:

These mixes were not homogeneous. While trying to solve workability issue, homogeneity problem was back to the mix. So, these trials were considered unsuccessful trials.

❖ Trial Mix# 17

Mixing details:

In this trial mix, the following question had been tried to be answered. What amount of additional water content (other than the water contained in sodium silicate solution and in sodium hydroxide solution) would make such new concrete mix homogeneous and workable enough? Mix# 16 was the reference mix for this trial mix.

An amount of 20 liters of water was added. Then, several multiple of 20 liters were added till a homogeneous and workable mix was prepared to the mix. This happened at 180 liters of additional water.

Observations:

Even though the mix was homogeneous and workable, it was looking sandy more than conventional concrete mixes and thus it was highly expected to have much less compressive strength.

❖ Trial Mix# 18

Mixing details:

In this mix, the ratio of ($\text{Na}_2\text{SiO}_3/\text{NaOH}$) was reduced further to be (1.67) instead of (2.00) in mix# 16. It is important to keep in mind that no additional water was added to

this mix other than the water contained in the sodium silicate and sodium hydroxide solutions. Neither superplasticizer nor stabilizer were added to the mix.

Observations:

Fortunately, separation of mixing ingredients was not observed and the mix was very homogeneous. Thankfully and unlike all previous mixes, without adding additional water or superplasticizer, the mix was workable and no major difficulties were faced in placing the mix in the molds.

Table 3.10 summarizes the data pertaining to the trial mixtures with CKD.

Table 3.10: CKD Trial Mixes Summary Data

Mix. No.	Geo-material	Curing Temp. (°C)	Powder (kg/m ³)	Water (Liter)	Superplasticizer (Liter)	Na ₂ SiO ₃ (kg/m ³)	water in Na ₂ SiO ₃ (Liter)	NaOH 16M (kg/m ³)	water in NaOH (Liter)	Water / Powder	Liquid / Powder	Alkali / Powder	Na ₂ SiO ₃ / NaOH	Observations	Average Compressive Strength (MPa)
1	CKD	60	400	0	0	150	93.8	57	34.8	0.32	0.52	0.52	2.63	Not Homogeneous	---
2	CKD	60	400	0	0	225	140.6	86	52.5	0.48	0.78	0.78	2.62	Not Workable	---
3	CKD	60	400	0	0	250	156.3	95	58.0	0.54	0.86	0.86	2.63	Not workable	---
4	CKD	60	400	0	0	260.9	163.1	99.1	60.5	0.56	0.90	0.90	2.63	Not Workable	---
5	CKD	60	400	0	3	225	140.6	86	52.5	0.48	0.78	0.78	2.62	Not workable	---
6	CKD	60	400	0	6	225	140.6	86	52.5	0.48	0.78	0.78	2.62	Not workable	---
7	CKD	60	400	0	9	225	140.6	86	52.5	0.48	0.78	0.78	2.62	Not workable	---
8	CKD	60	400	0	12	225	140.6	86	52.5	0.48	0.78	0.78	2.62	Not workable	---
9	CKD	60	400	5	0	225	140.6	86	52.5	0.50	0.79	0.78	2.62	Not workable	---
10	CKD	60	400	10	0	225	140.6	86	52.5	0.51	0.80	0.78	2.62	Not workable	---
11	CKD	60	400	15	0	225	140.6	86	52.5	0.52	0.82	0.78	2.62	Not workable	---
12	CKD	60	400	20	0	225	140.6	86	52.5	0.53	0.83	0.78	2.62	Not workable	---
13	CKD	60	400	0	0	138.1	86.3	69.1	42.2	0.32	0.52	0.52	2.00	Not Homogeneous	---
14	CKD	60	400	0	0	186.6	116.6	93.3	56.9	0.43	0.70	0.70	2.00	Not Homogeneous	---
15	CKD	60	400	0	0	211	131.9	105.4	64.3	0.49	0.79	0.79	2.00	Not Homogeneous	---
16	CKD	60	400	0	0	240	150.0	120	73.2	0.56	0.90	0.90	2.00	Not Homogeneous	---
17	CKD	60	400	180	0	240	150.0	120	73.2	1.01	1.35	0.90	2.00	Homogeneous & Workable	4.0
18	CKD	60	400	0	0	225	140.6	134.7	82.2	0.56	0.90	0.90	1.67	Homogeneous & Workable	17.7

3.2.4: Trial Mixes with LSP

Similar to the way that CKD trials were mixed and checked for their tenancy to act as the main binding material in preparing new concrete mixes, LSP was the second potential candidate that was investigated.

Mixing ingredients of LSP mixes were LSP powder, #4 coarse aggregates (4.75 mm), #8 coarse aggregates (2.36 mm), fine aggregates, water, sodium hydroxide solution, and sodium silicate solution.

Curing temperature (at oven) was fixed to be (60° C) for 24 hours. LSP content was fixed at 400 kg/m³. A 3:2 ratio of coarse to fine aggregates was fixed and used along all experiments as well as a 9:1 ratio of #4 to #8 coarse aggregates.

Mixing details:

In LSP trial mixes, mixing was started for mix# 1 with the same configuration and ingredients of the best mix in CKD trials except that CKD was replaced by LSP while all other parameters were kept unchanged. Then, several parameters were changed in preparing LSP trials. These parameters included curing temperature, alkali to powder ratio, and sodium silicate to sodium hydroxide ratio. The details of LSP trial mixes are summarized and tabulated in Table 3.11 which includes mixing ingredients, observations, and compressive strength values.

Observations:

It was observed that all LSP trial mixes did not suffer a problem in homogeneity nor workability. Despite this positive observation, it was looking sandy more than

conventional concrete mixes and all compressive strength results were extremely low
not reaching (7 MPa) for all trials-

Table 3.11: LSP Trial Mixes Summary Data

Mix. No.	Geo-material	Curing Temp.	powder	Na ₂ SiO ₃	NaOH	water in NaOH	Water / Powder	liquid / Powder	Alkali / Powder	Na ₂ SiO ₃ / NaOH	Observations	Average Compressive Strength (MPa)
1	LSP	60	400	225	135	82.3	0.56	0.90	0.90	1.67	homogeneous & workable	5.6
2	LSP	24	400	225	135	82.3	0.56	0.90	0.90	1.67	homogeneous & workable	2.0
3	LSP	60	400	192	115	69.9	0.47	0.77	0.77	1.67	homogeneous & less workable than mix# 1 workable	4.0
4	LSP	60	400	240	120	73.2	0.56	0.90	0.90	2.00	Same as 15	4.6
5	LSP	60	400	257	103	62.7	0.56	0.90	0.90	2.50	homogeneous & very workable	6.7
6	LSP	60	400	270	90	54.9	0.56	0.90	0.90	3.00	homogeneous & workable	5.2
7	LSP	60	400	286	114	69.7	0.62	1.00	1.00	2.50	homogeneous & very workable	6.4
8	LSP	60	400	343	137	83.6	0.74	1.20	1.20	2.50	homogeneous & very workable	5.3
9	LSP	24	400	286	114	69.7	0.62	1.00	1.00	2.50	homogeneous & very workable	2.2

3.2.5: Trial Mixes with BHD

Similar to the way that CKD trials and LSP trials were investigated for their tenancy to act as the main binding material in preparing new concrete mixes, BHD was the third potential candidate that was investigated.

Mixing ingredients of BHD mixes were BHD powder, #4 coarse aggregates (4.75 mm), #8 coarse aggregates (2.36 mm), fine aggregates, water, sodium hydroxide solution, and sodium silicate solution.

Curing temperature (at oven) was fixed to be (60° C) for 24 hours. BHD content was fixed at 400 kg/m³. A 3:2 ratio of coarse to fine aggregates was fixed and used along all experiments as well as a 9:1 ratio of #4 to #8 coarse aggregates.

Mixing details:

In BHD trial mixes, alkali to powder ratio was changed in preparing the trials while sodium silicate to sodium hydroxide ratio was kept almost constant. No additional water was separately added to the mixes. The details of BHD trial mixes are summarized and tabulated in table 3.12 which includes mixing ingredients, observations, and compressive strength values.

Observations:

It was observed that all BHD trial mixes did not suffer a problem in homogeneity nor workability except for the last mix. Also, all compressive strength results were extremely low with maximum achieved value of (9 MPa).

Table 3.12: BHD Trial Mixes Summary Data

Mix. No.	Geo-material	Curing Temp.	powder	Na ₂ SiO ₃	NaOH	water in NaOH	Water / Powder	Liquid / Powder	Alkali / Powder	Na ₂ SiO ₃ / NaOH	Observations	Average Compressive Strength (MPa)
1	BHD	60	400	285	117	71.2	0.62	1.00	1.00	2.44	homogeneous & Very workable	5.4
2	BHD	60	400	258	105	63.8	0.56	0.91	0.91	2.47	homogeneous & workable	7.4
3	BHD	60	400	229	94	57.3	0.50	0.81	0.81	2.43	homogeneous & workable	8.8
4	BHD	60	400	200	81	49.4	0.44	0.70	0.70	2.48	Less homogeneous & workable than previous one	9.0
5	BHD	60	400	171	69	42.1	0.37	0.60	0.60	2.48	Not homogeneous	----

3.3: MODIFIED MIXES STAGE

In this stage, investigations are going to take the results which were achieved in the previous mixing stage (Trial Mixes Stage) further through more specific and directed mixing methodology. The effect of each nonconventional ingredients in such new concrete type under development was monitored as well.

Moreover, the investigations that were carried out in this stage was to see if better concrete mixes can be prepared still without any contribution of ordinary Portland cement.

Another important aim of this stage was to have clearer picture and better understanding of the mixing environment, methodology, sequence of mixing, affecting parameters, and major mixing ingredients and their effects on such type of emerging concrete technology.

3.1.1: Summary of Previous Stage Achievements

The two main achievements are:

First: Further investigations will be carried out for CKD and stopped for LSP and BHD. This is because CKD was better than LSP and BHD to act as the main cementitious material instead of ordinary Portland cement as shown in details in the trial mixes stage.

Second: The best trial mix for CKD (i.e. trial mix# 18) will be the reference and starting mix in this stage.

3.1.2: Modified Mixes methodology

In this stage, and in order to see how changing mixing parameters will affect the results of the mixes under investigation, mixing methodology and sequence of ingredients mixing were done in accordance with the following points in which detailed procedures and mixing rearrangements will be illustrated:

- Mixing methodology and sequence were changed and reorganized. First, only dry ingredients (i.e. #4 coarse aggregate, #8 coarse aggregate, fine aggregate, and CKD) got mixed separately until they were all well mixed. Then, all wet ingredients (i.e. sodium hydroxide, sodium silicate, and any other liquid ingredients (i.e. water and/or superplasticizer) if any) got mixed separately. Finally, the two separate mixes (i.e. dry and wet) got mixed together to form the final concrete trial mix. This procedure was followed in each and every mix that was being prepared in this stage.

- Continuing with trial mix#18 (with (0.90) alkali / powder ratio and (1.67) Na_2SiO_3 / NaOH ratio), this mixing stage was started by making sure that this mix was working by repeating this trial and recall it (mix# M-001) in this stage of investigations.
- Then, by lowering the ratio of Na_2SiO_3 / NaOH while keeping the same best Alkali/Powder ratio achieved constant, the following mixes were prepared:
 - mix# M-102
 - mix# M-103
 - mix# M-104
 - mix# M-105
- Then, by raising the ratio of Na_2SiO_3 / NaOH while keeping the same best Alkali/Powder ratio achieved constant, the following mixes were prepared:
 - mix# M-206
 - mix# M-207
 - mix# M-208
 - mix# M-209
 - mix# M-210
 - mix# M-211
- This was done to see the effect of these two major ingredients in the mix with keeping the water content almost constant.
- Since raising the ratio of (Na_2SiO_3 / NaOH) resulted in mixes with higher compressive strength, as it will be seen later in this study, but to a certain value of this ratio the mixes were suffering from workability issues till a point where the mixes were not workable at all. So, finding a way to make the mix more workable was necessary.

- Although adding water was not an effective solution to solve workability, adding water was done to the mix at first. This was only to confirm previous observations and results. Mix# M312 was prepared.

Second, Superplasticizers were tried to see if they can help in workability. We prepared the following mixes by adding some amounts of superplasticizer to “mix# M-211”:

- mix# M-313
 - mix# M-314
 - mix# M-315
 - mix# M-316
 - mix# M-317
-
- After that, the ratio of (Alkali / Powder) was changed while keeping the same best ratio of (Na_2SiO_3 / NaOH) which achieved the highest compressive strength with in (mix# M-209), the following mixes were prepared:
 - mix# M-418
 - mix# M-419
 - mix# M-420
 - mix# M-421
 - mix# M-422
 - mix# M-423

The data pertaining to the modified mixes is presented in Table 3.13.

Table 3.13: Modified mixes summary data

Mix. No.	Geo-material	Curing Temp. (°C)	Powder (Na ₂ SiO ₃)	Water (Liter)	Superplasticizer (Liter)	Na ₂ SiO ₃ (kg/m ³)	water in Na ₂ SiO ₃ (Liter)	NaOH 16M (kg/m ³)	water in NaOH (Liter)	Water / Powder	Liquid / Powder	Alkali / Powder	Na ₂ SiO ₃ / NaOH	Mixing Notes / Details	Testing Age (H)	Average Compressive Strength (MPa)
M-001	CKD	60	400	0	0	225	140.6	134.7	82.2	0.56	0.90	0.90	1.67		72 (3)	15.7
M-102	CKD	60	400	0	0	210	131.3	150	91.5	0.56	0.90	0.90	1.40	better workability than M-001	24 (1)	16.7
M-103	CKD	60	400	0	0	196.4	122.8	163.6	99.8	0.56	0.90	0.90	1.20	almost self-consolidated	48 (2)	17.5
M-104	CKD	60	400	0	0	180	112.5	180	109.8	0.56	0.90	0.90	1.00	hardly placed in the mold (lost workability)	48 (2)	17.5
M-105	CKD	60	400	0	0	160	100.0	200	122.0	0.56	0.90	0.90	0.80	NOT WORKABLE	----	----
M-206	CKD	60	400	0	0	236.6	147.9	124.6	76.0	0.56	0.90	0.90	1.90	Very good workability	72 (3)	19.9
M-207	CKD	60	400	0	0	244.7	152.9	116.5	71.1	0.56	0.90	0.90	2.10	good workability	72 (3)	21.1
M-208	CKD	60	400	0	0	250.9	156.8	109.1	66.6	0.56	0.90	0.90	2.30	workability was less than previous mix	48 (2)	23.4
M-209	CKD	60	400	0	0	257.1	160.7	102.9	62.8	0.56	0.90	0.90	2.50	workability was less than previous mix	24 (1)	24.7
M-210	CKD	60	400	0	0	262.7	164.2	97.3	59.4	0.56	0.90	0.90	2.70	stick to mold quickly - not allowing to pour	----	----
M-211	CKD	60	400	0	0	267.7	167.3	92.3	56.3	0.56	0.90	0.90	2.90	Not Workable	----	----

Table 3.13 (continued): Modified mixes summary data

Mix. No.	Geo-material	Curing Temp. (°C)	Powder (kg/m ³)	Water (Liter)	Superplasticizer (Liter)	Na ₂ SiO ₃ (kg/m ³)	water in Na ₂ SiO ₃ (Liter)	NaOH 16M (kg/m ³)	water in NaOH (Liter)	Water / Powder	Liquid / Powder	Alkali / Powder	Na ₂ SiO ₃ / NaOH	Mixing Notes / Details	Testing Age (H)	Average Compressive Strength (MPa)
M-312	CKD	60	400	40	0	267.7	167.3	92.3	56.3	0.66	1.00	0.90	2.90	Not Workable	----	----
M-313	CKD	60	400	0	4	267.7	167.3	92.3	56.3	0.56	0.91	0.90	2.90	Not Workable	----	----
M-314	CKD	60	400	0	8	267.7	167.3	92.3	56.3	0.56	0.92	0.90	2.90	Not Workable	----	----
M-315	CKD	60	400	0	16	267.7	167.3	92.3	56.3	0.56	0.94	0.90	2.90	Not Workable	----	----
M-316	CKD	60	400	0	24	267.7	167.3	92.3	56.3	0.56	0.96	0.90	2.90	Not Workable	----	----
M-317	CKD	60	400	0	48	267.7	167.3	92.3	56.3	0.56	1.02	0.90	2.90	Not Workable	----	----
M-418	CKD	60	400	0	0	228.6	142.9	91.4	55.8	0.50	0.80	0.80	2.50	Takes long time to get homo (>10 min). Not Workable	----	----
M-419	CKD	60	400	0	0	200.0	125.0	80.0	48.8	0.43	0.70	0.70	2.50	Not Homogeneous	----	----
M-420	CKD	60	400	40	0	200.0	125.0	80.0	48.8	0.53	0.80	0.70	2.50	Not Homogeneous	----	----
M-421	CKD	60	400	80	0	200.0	125.0	80.0	48.8	0.63	0.90	0.70	2.50	Homogeneous - But looks sandy	72 (3)	10.1
M-422	CKD	60	400	0	0	285.7	178.6	114.3	69.7	0.62	1.00	1.00	2.50	Homogeneous & workable	24 (1)	21.0
M-423	CKD	60	400	0	0	314.3	196.4	125.7	76.7	0.68	1.10	1.10	2.50	Homogeneous & workable	24 (1)	17.5

3.4: DETAILED TESTING STAGE

Detailed tests were performed on the best mix, i.e. Mix# M-209, which has been achieved in previous section. These tests will provide us with better understanding of the new concrete type under development and investigation. This stage revealed more information about this alkali-activated concrete which will greatly help to understand mechanical properties, and durability characteristics of the developed concrete.

The followings are the tests (Table 3.14) which were performed on the best alkali-activated concrete mix achieved in this study:

- Compressive strength
- Flexure strength
- Modulus of elasticity
- Scanning Electron Microscopy (SEM)
- Energy Dispersive Spectroscopy (EDS)
- Chemical Composition
- Reinforcement Corrosion
- Sulfate Attack

Table 3.14: Performed tests details and reference standards

Test Type	Samples Size	Curing Period	Age of testing	# of Specimens	Solution Required	Test Standard
Compressive strength	50x50x50 mm <u>Cube</u>	1 day	1, 3, 7, 28, 90 days	15	-	ASTM C39
Flexural Strength	40x40x160 mm <u>Bar</u>	1 day	7 days	3	-	ASTM C293
Modulus of Elasticity	3x6 " <u>Cyl.</u>	1 day	7 days	3	-	ASTM C469
Reinforcement Corrosion	3x6 " <u>Cyl.</u>	1 day	every 4 weeks	3	5% (NaCl)	ASTM C876 (SCE)
Sulfate Attack	50x50x50 mm <u>Cube</u>	1 day	90 - 180 - 270 days	3	2.1% (NaSO ₄ + MgSO ₄)	ASTM C1012

3.4.1: Compressive strength

Compressive strength test is the most common test on hardened concrete. This is because, normally, most of concrete characteristics are related to the compressive strength. In addition to that, it is a simple and easy to do test.

Specimens were cast in 50x50x50 cast-iron molds. Molds were prepared by assembling all components of each mold together (base, sides, and dividers) and tighten them well to prevents mortar leakage. Then the mold was covered with oil to prevent the

development of bond between the mold and the specimen. Meanwhile, concrete mix was prepared as per the best way achieved in the modified mixing stage.

Then, the mix was placed in the molds under the effect of the vibrating table. After that, the mix was placed in the oven for curing. 24 hours later, the specimens were demolded, labeled, and prepared for testing or kept in the LAB till testing time.

Table 3.15: Compressive Strength Samples Labeling

Age of testing (Day)	1			3			7			28			90		
Specimen Labeling	S11	S12	S13	S21	S22	S23	S31	S32	S33	S41	S42	S43	S51	S52	S53

3.4.2: Flexure strength

Flexure strength (also known as modulus of rupture or bend strength) is the flexure stress in concrete just before it yields. This test provide us with the highest flexural stress experienced in concrete. Three-point flexural test was used to get the specimen strength in flexure. Set up is illustrated in Figure 3.7. The main advantage of this test type is the ease of the specimen preparation and testing. However, this method has also some disadvantages: the results of the testing method are sensitive to specimen and loading geometry.

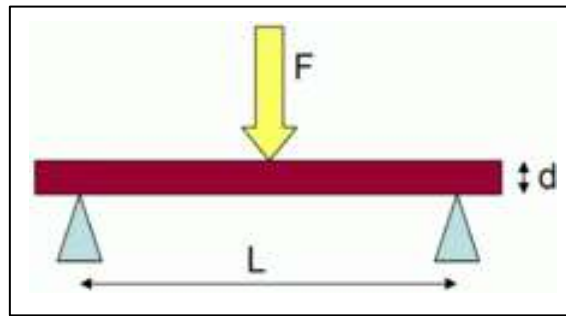


Figure 3.7: FBD of Three-point Flexure Test

Specimens were cast in 40x40x160 mm molds. Molds were prepared by assembling all components of each mold together and tighten them well to prevent mortar leakage. Then, mold surface was covered with oil to prevent the development of bond between the mold and the specimen. Then, the mix was placed in the molds under the effect of the vibrating table.

After that, the mix was placed in the oven for curing. 24 hours later, the specimens were demolded, labeled, and prepared for testing. Four samples were prepared. They were labeled (S-91, S-92, and S-93).

Testing gage records load value (F) and corresponding beam transverse extension (deflection under loading). The load was continuously increased till the beam was not able to bare more load (recording maximum force values). This value was used to calculate the flexural strength in terms of stress using the following equation:

$$(\sigma = 3FL / 2bd^2)$$

- F is the load (force) at the fracture point (N)
- L is the length of the support span = 160mm
- b is width = 40mm
- d is thickness = 40mm

For each one of the three tested samples all recorded values of loads and corresponding extensions are presented in appendix-C.

3.4.3: Modulus of elasticity

Modulus of Elasticity (also known as the elastic modulus or Young's modulus) measures the stiffness of a specific material (I.e. the ability of the material to resist deformation elastically under applied load). The higher the elastic modulus of a material, the stiffer this material will be. Generally, the modulus is represented as the stress over strain:

$$\lambda \stackrel{\text{def}}{=} \frac{\text{stress}}{\text{strain}}$$

Specimens were cast in 3x6'' cylindrical molds. Molds were prepared by assembling all components of each mold together and tighten them well to prevent mortar leakage. Then, mold surface was covered with oil to prevent the development of bond between the mold and the specimen. Then, the mix was placed in the molds under the effect of the vibrating table.

After that, the mix was placed in the oven for curing. 24 hours later, the specimens were demolded, labeled, and kept in room temperature. 7 days later, specimens were tested. Three samples were prepared and labeled as (S-101, S-102, and S-103).

The testing procedure as described in ASTM C469 standard test [25] was followed in determining the modulus of elasticity in compression in this study. In this procedure, slowly increasing compression load (and hence compressive stress) was applied to each specimen. With the help of the strain gages attached to the specimens, longitudinal strains were detected and saved to the attached computer machine. Applied loads and longitudinal strains were recorded and presented in appendix-D. The test was terminated when the applied load achieves 40% of the specimen compressive stress which 45 kN for our case (resulting in 10 MPa). Then, stress-strain curve was plotted and the modulus of elasticity was calculated as the slope of the straight line. In the next chapter, results are fully presented and discussed. Moreover, the experimental value was compared with the theoretical value calculated using the following equation from the ACI-318 Code:

$$w_c^{1.5} 0.043 \sqrt{f'_c} \quad (\text{in MPa})$$

3.4.4: Scanning Electron Microscopy (SEM)

Both microstructure and chemical compositions of concrete control its properties. Since microstructure of concrete depends on how concrete is made, it provides more control on over chemical structure. In other words, concrete behavior is strongly affected by how it is made and thus studying concrete microstructure helps greatly to predict, relate, explain, control, and improve concrete properties.

Especially in preparing new concrete or improving an existing concrete types (which was the case in this study), besides empirical research, microstructure investigations provide deeper understanding and more specific explanations on properties changes. This will eventually facilitate and speed up the way to improve and achieve better materials.

Basically, Morphology is the study of how things are formed or made. Scanning electron microscopy (SEM) technique was used to study the morphology of the prepared new concrete type. At a very high magnification, SEM allow for deeper investigation of the sample surface (and near surface). It is a very powerful tool to study matter's morphology and investigate its microstructure. Electron microscope is used instead of optical ones providing us with the advantage that electrons have much shorter wavelength than photons and thus allowing to observe and study the specimens at the atomic resolution. Secondary electron vivid Images, product of SEM technique, provide information about the specimens' surface and near surface topography that is scanned with electron beams [28].

Small pieces of the broken specimens in the compression test that was performed on our concrete in section 3.4.1 were used as the samples to produce SEM images to investigate the morphology of our concrete. Electron images were captured using a

specialized software that was equipped with the electron spectrometer at the characterization facility at KFUPM Research Institute.

Five electron images were captured and presented in the next chapter (Results Chapter). Images were captured at different resolutions allowing to discover and go through the microstructure more and more. Investigations of specimens' morphology reaches to the level of microns by such techniques being utilized.

3.4.5: Energy Dispersive Spectroscopy (EDS)

Qualitative analysis was performed to find what elements are forming the specimen under investigation. Energy dispersive spectroscopy (EDS) is used for this purpose since a complete spectrum can be obtained in a short period of time. In energy dispersive analysis, X-ray spectrum is generated for the scanned area of the SEM showing what elements are present. Using tables of energies, elements are identified lines in the X-ray spectrum as will be seen in the next chapter (where the spectra will be presented and discussed).

Emitted X-ray energy levels are unique for each element. This allows for clear identification of most of the elements present in the sample. A detector is used to separate the characteristic X-rays for each one of the different element in the generated spectrum. Then, the quantity of each specific element is reported using a software that is incorporated with the EDS system.

In qualitative analysis of specimens, an element is said to be major component of the sample being analyzed if it is more than 10% of the sample by weight and minor if it is available with 1 to 10% by weight. Any other component that is present with a weight percentage less than 1% is said to be a trace element. EDS is also used for quantitative

analysis. The atomic percentage of the various chemical compositions that forms the sample under investigation is derived.

The obtained X-ray spectrum shows the number of counts, received by the detector) per Electron Volt (eV) on the y-axis and the energy level of theses counts in kilo Electron volt (keV) on the x-axis. The number of counts and the atomic percentage are proportionally related in a way or another.

3.4.6: Reinforcement Corrosion

Corrosion of steel reinforcement is one of the major issue that **MUST** be considered when dealing with concrete especially in hot and severe conditions (like the conditions exist in our region). Corrosion was investigated by continuously monitoring the testing specimens which were placed in the NaCl solution and measuring both potential difference as well as concrete current density, i.e. I_{cor} . This process was performed for a period of 270 days. Then, the recorded values were compared with reference values in the relevant standard.

Specimens were cast in 3x6'' cylindrical molds. Molds were prepared by assembling all components of each mold together and tighten them well to prevent mortar leakage. Then, mold surface was covered with oil to prevent the development of bond between the mold and the specimen. Steel bar was placed in the center of the cylindrical mold. Then, the mix was placed in the molds under the effect of the vibrating table.

After that, the mix was placed in the oven for curing. 24 hours later, the specimens were demolded, labeled, and kept in room temperature. Three samples were prepared and labeled as (S-71, S-72, and S-73).

7 days later, 0.85 molar NaCl solution was prepared and the specimens were put in the solution. The solution was prepared according to the following formula:

$(\text{NaCl})_{5\%} = \text{"1000"} \text{ grams of water} + \text{"50"} \text{ grams of Sodium chloride}$

3.4.7: Sulfate attack test

Sulfate attack is a problem that the concrete faces especially elements which are exposed to soil that is rich in sulfate. It is an important measure of concrete quality. The more that the concrete prevents sulfate from attacking the element, the better is the concrete.

Specimens were cast in 50x50x50 cast-iron molds and were placed in the oven for curing. 24 hours later, the specimens were demolded, labeled, and kept in room temperature. Three samples were prepared and labeled as (S-81, S-82, and S-83). 7 days later, a 2.1 % ($\text{NaSO}_4 + \text{MgSO}_4$) solution was prepared and all of the three specimens were placed in this solution (without any coating).

Specimens were exposed in the solution for 270 days. Sulfate resistance was investigated three times (90, 180, and 270 days) by both:

- Visual examination
- Weight Loss

CHAPTER 4

RESULTS

4.1: BEHAVIOUR OF THE MIXES

Effect of (Na_2SiO_3 / NaOH) Ratio on Compressive Strength:

One of the main parameters that was affecting this type of concrete mixes was the ratio of “ Na_2SiO_3 / NaOH”. Investigations of mixes prepared in this study indicated that the ratio of “ Na_2SiO_3 / NaOH” and compressive strength were strongly related to each other. Compressive strength values and corresponding “ Na_2SiO_3 / NaOH” ratios are summarized in Table 4.1:

Table 4.1: Compressive Strength Vs (Na_2SiO_3 / NaOH)

(Na_2SiO_3 / NaOH) Ratio	(Water / Powder) Ratio	Compressive Strength (MPa)
1.00	0.56	17.5
1.20	0.56	17.5
1.40	0.56	16.7
1.67	0.56	15.7
1.90	0.56	19.9
2.10	0.56	21.1
2.30	0.56	23.4
2.50	0.56	24.7

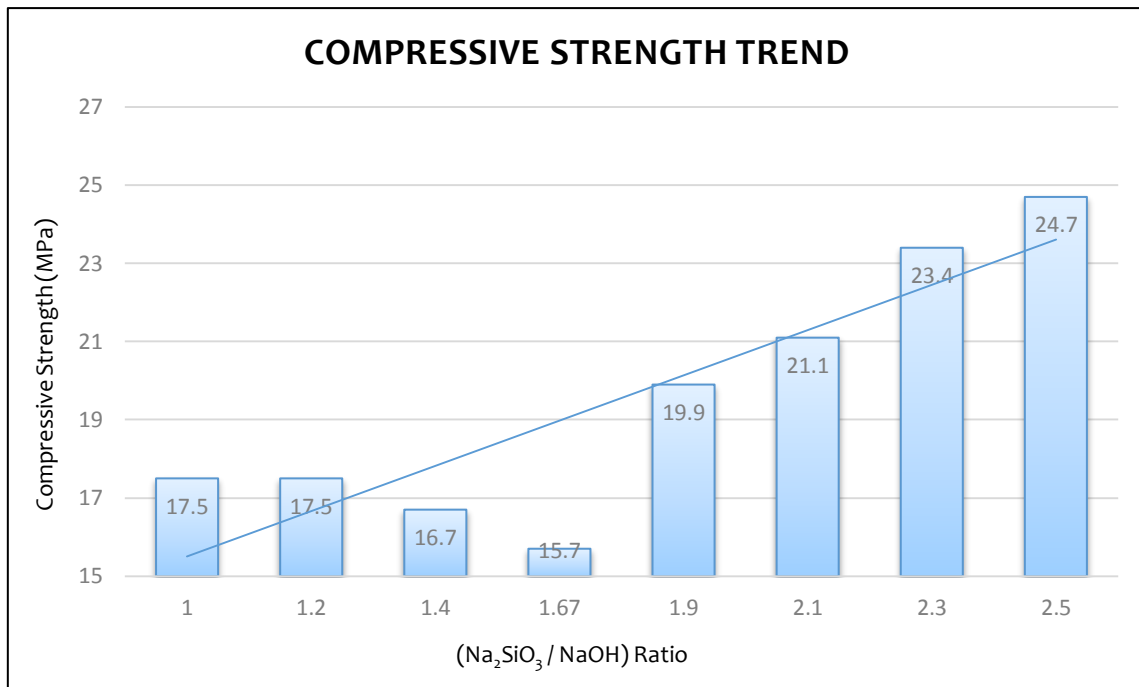


Figure 4.1: Compressive Strength Trend

In general, higher ratios were resulted in higher compressive strength. This was possibly because of silica content effect. In other words, as this ratio gets higher, the silica content gets higher which is a major source of providing strength to concrete since it typically acts as a binding agent in the concrete. As seen in Figure 4.1, in which this relation is plotted, clearly an increasing trend can be observed.

Effect of $(\text{Na}_2\text{SiO}_3 / \text{NaOH})$ on Homogeneity and Workability:

As the ratio of “ $\text{Na}_2\text{SiO}_3 / \text{NaOH}$ ” decreased (from 1.67 to 0.80), the mix workability improved. But workability was gradually lost till a non-workable mix at all was resulted at 0.80 sodium silicate to sodium hydroxide ratio. Also, as the same ratio increased, mixes started to get harder gradually until, at a very high ratios of “ $\text{Na}_2\text{SiO}_3 / \text{NaOH}$ ” at 2.7 and 2.9, mixes were no more workable. It is important to notice that

“water/powder” was kept almost constant to avoid varying more variables and thus leading to not a very clear picture of what was going on with the mixes. Table 4.2 summarizes all these values and observation of homogeneity and workability.

Table 4.2: Homogeneity and Workability Observations Summary

(Na₂SiO₃ / NaOH) Ratio	Homogeneity	Workability
0.80	Homogeneous mix	Not workable at all
1.00	Homogeneous mix	hardly placed in the mold (rapid loss of workability)
1.20	Homogeneous mix	Excellent workability and flowability
1.40	Homogeneous mix	Very good workability
1.67	Homogeneous mix	Good workability
1.90	Homogeneous mix	Very good workability
2.10	Homogeneous mix	good workability
2.30	Homogeneous mix	good workability
2.50	Homogeneous mix	Limited time to place (quickly lost workability)
2.70	Homogeneous mix	Not workable at all - stuck to mixing bowl quickly - not allowing to pour
2.90	Barely homogeneous mix	Not workable at all – hardened even before it gets well mixed

Effect of Water Content:

Adding water to such type of emerging concrete mixes was not an effective solution. It can be observed that the amount of water added independently, i.e. not part of any chemical solution (**Na₂SiO₃ or NaOH**), helped slightly to improve workability but it was required to add a very high amount. As seen in the data summarized in Table 4.3,

even with 10% of powder content extra water (40 liters extra water) was not enough helping even to pour concrete and it was rapidly stuck to the mixing bowl.

Additional water was not a choice to solve workability for two main reasons:

First: Huge water content was needed to reach workable mixes. This was clear since a “0.1” increase of water to powder ratio to become “0.66” did not lead to a workable mix.

Second: Adding big amount of water will severely affect the strength of the mixes. See values of the last two in Table 4.3.

Table 4.3: Homogeneity and Workability Observations Summary

water in Na ₂ SiO ₃ (Liter)	water in NaOH (Liter)	Before Additional Water	Add. Water (Liter)	After Additional Water	Total Water (Liters)	Water/Powder Ratio	Compressive Strength (MPa)
140.6	52.5	Not workable	5	Not workable	198.1	0.5	---
140.6	52.5	Not workable	10	Not workable	203.1	0.51	---
140.6	52.5	Not workable	15	Not workable	208.1	0.52	---
140.6	52.5	Not workable	20	Not workable	213.1	0.53	---
125.0	48.8	Not homogeneous	40	Not homogeneous	213.8	0.53	---
167.3	56.3	Not workable	40	Not workable	263.6	0.66	---
125.0	48.8	Not homogeneous	80	homogeneous and workable But looks sandy	253.8	0.63	10.1
150.0	73.2	Not homogeneous	180	Homogeneous & workable	403.2	1.01	4.0

Effect of Superplasticizer:

Sugar-based superplasticizer “Sika ViscoCrete-10 R” did not help at all to improve workability even with very high dosages, i.e. reaching up to 6 times the recommended maximum dosage by manufacturer. Note that the maximum limits specified by the

manufacturer is for producing self-compacting concrete for concrete mixes made using ordinary Portland cement. Data is summarized in Table 4.4.

Table 4.4: Superplasticizer Data Summary

Superplasticizer (Liter)	Observations
3	Not workable – mix hardened very rapidly and stuck to mixing bowl
6	Not workable – no improvement in workability
9	Not workable – no improvement in workability
12	Not workable – no improvement in workability

Effect of (Alkali / Powder) Ratio:

As the ratio of (Alkali / Powder) increased, the mix homogeneity improved. The same was for workability. Homogeneity was probably improved because of the increased amount of alkalis in which silica is contained, main binding agent, as well as sodium hydroxide as an activator for sodium silicate. The resulted improvements in workability were also reasonable since, implicitly, water content increased as alkalis are in water based solution forms. See Table 4.5, in which related data is summarized.

Table 4.5: (Alkali / Powder) Data Summary

(Alkali / Powder) Ratio	Homogeneity	Workability	Compressive Strength (MPa)
0.7	Not homogeneous mix	-----	-----
0.8	Takes long time to get homogeneous (more than 10 min of mixing).	Not Workable	-----
0.9	homogeneous mix	Workable mix	24.7
1.0	homogeneous mix	Better workable mix than previous	21.0
1.1	homogeneous mix	Better workable mix than previous	17.5

Despite the improvements in mix homogeneity and workability, compressive strength was majorly affected which was decreasing as the ratio of “alkali / powder” increases.

See Figure 4.2 in which these results are plotted.

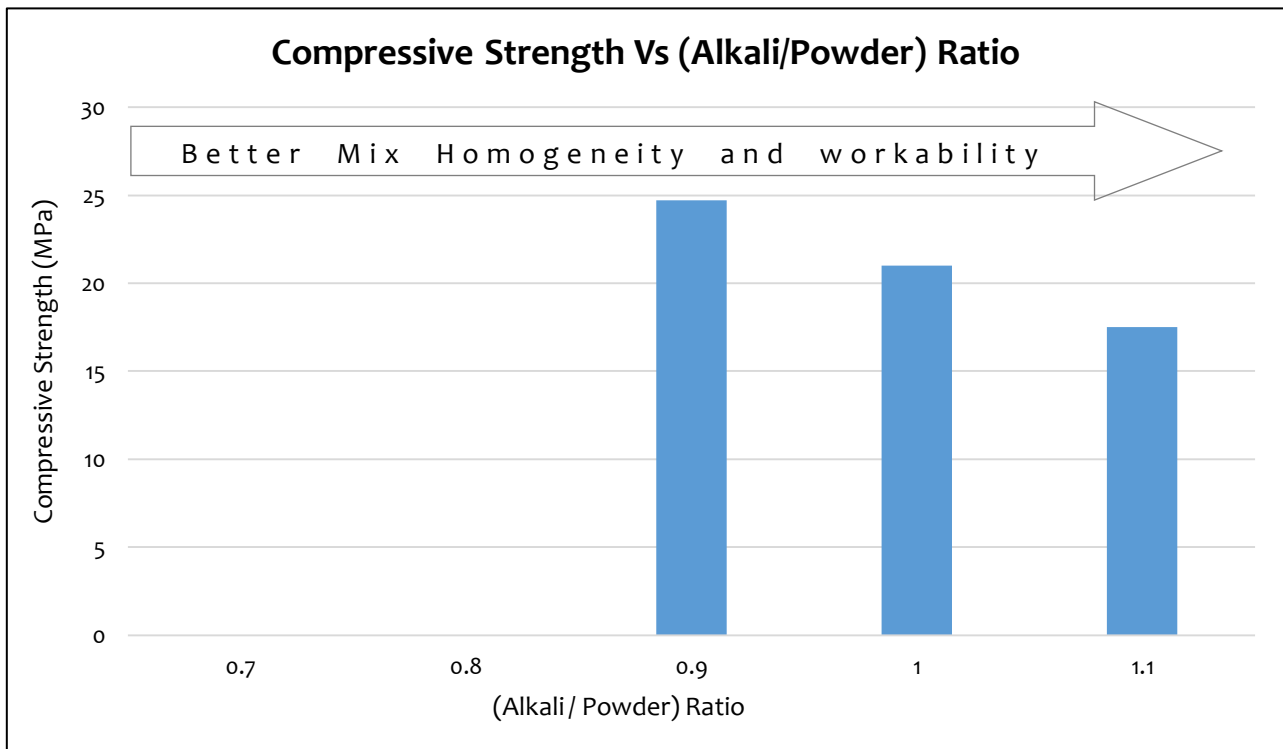


Figure 4.2: Compressive Strength Vs (Alkali/Powder) Ratio

4.2: MECHANICAL PROPERTIES

Good concrete always possess good mechanical properties. Because of that, the first and most important results that should be looked at are the results that clarify the mechanical properties, especially when developing new concrete type. These properties includes; compressive strength, flexural strength, and modulus of elasticity.

Compressive strength tests performed on the prepared concrete specimens resulted a very good compressive strength. The compressive strength was resulted to be as high as (25 MPa). This is a very good value especially for such type of concrete that was prepared without Portland cement at all. Compressive strength test results are tabulated in Table 4.6 for all specimens tested 1, 3, 7, 28, and 90 days later after 24 hours curing at 60 °C in the oven. Also, these results are illustrated in Figure 4.3 as well.

Table 4.6: Compressive Strength Test Results

Age of testing	1 day			3 days			7 days			28 days			90 days		
Specimen Labeling	S11	S12	S13	S21	S22	S23	S31	S32	S33	S41	S42	S43	S51	S52	S53
Compressive strength (MPa)	24.4	24.1	25	24.7	24.1	24.1	24.9	24.4	24.2	24.4	24.5	25.2	24.9	24.8	25.3
Average Compressive strength (MPa)	24.5			24.3			24.5			24.7			25.0		

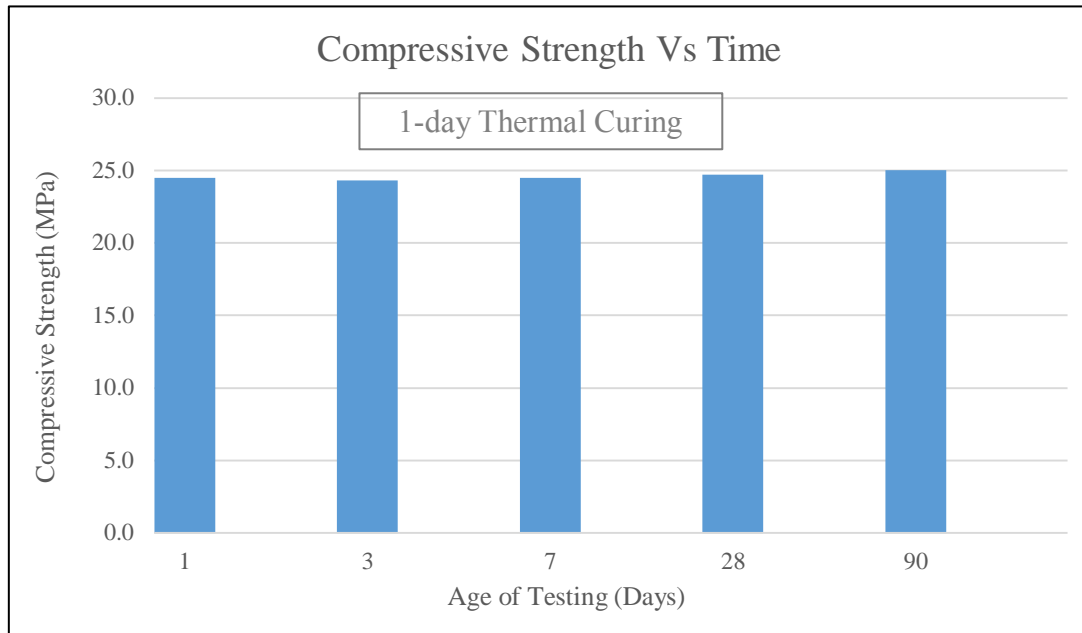


Figure 4.3: Compressive Strength Results

Flexural strength was another important property which has been investigated as per the procedure explained in section 3.4.2. Utilizing the resulting data attached in Appendix-C and the equation in section 3.4.2, flexural strength (f_r) was calculated and the resulting stress is shown in Figure 4.4.

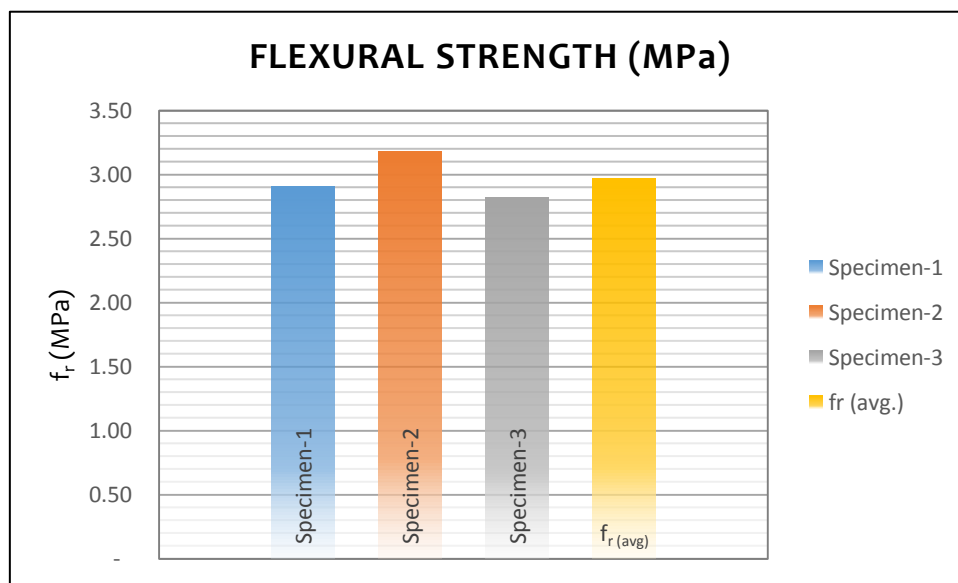


Figure 4.4: Flexural Strength Results

The results were very reasonable and strongly related to values that the ACI Code specifies for conventional concrete. According to the ACI-318 code, flexural strength was predicted using the following equation:

$$f_r = 0.62\lambda\sqrt{f'_c}$$

Comparing the results of actual values with the ones calculated using this equation, a calculated corresponding compressive stress was calculated to be (22.95 MPa) to the average flexural stress for the tested specimens of (2.97 MPa). The calculated corresponding compressive stress was very close to the actual value tested in the LAB of compressive strength that was obtained earlier in the compressive strength tests results of our concrete specimens in this study.

As per the procedure explained in section 3.4.3, stress-strain curve was plotted (see Figure 4.5) using the data attached in Appendix-D.

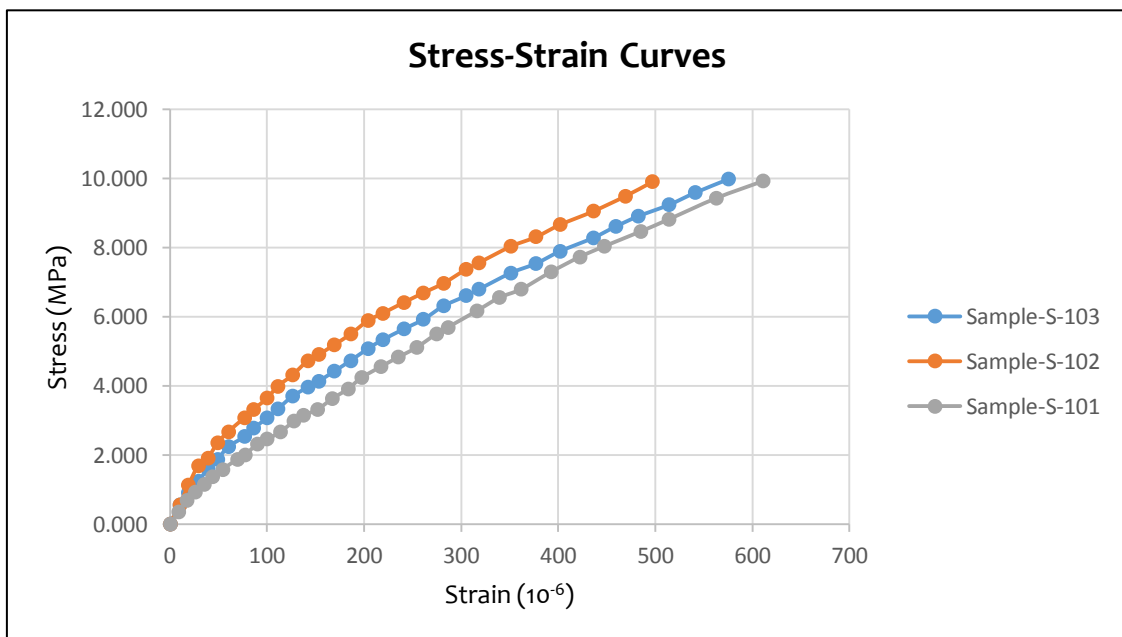


Figure 4.5: Stress-Strain Curves for Tested Specimens

From Figure 4.5, modulus of elasticity (E) was calculated for the three tested specimens to be (16.4 GPa), (20.1 GPa), and (17.4 GPa) for sample S-101, S-102, and S-103 respectively as tabulated in Table 4.7. These values were lower than the values which the ACI-318 equation mentioned earlier at section 3.4.3 gives for ordinary concrete. See Figure 4.6 for comparison between the values of E for the tested specimens with the calculated one using the equation provided in ACI-318.

Table 4.7: Tested and calculated Modulus of Elasticity

	Sample-S-101	Sample-S-102	Sample-S-103	E Average	E _{ACI} (MPa)
E (GPa)	16.4	20.1	17.4	18.0	26.5

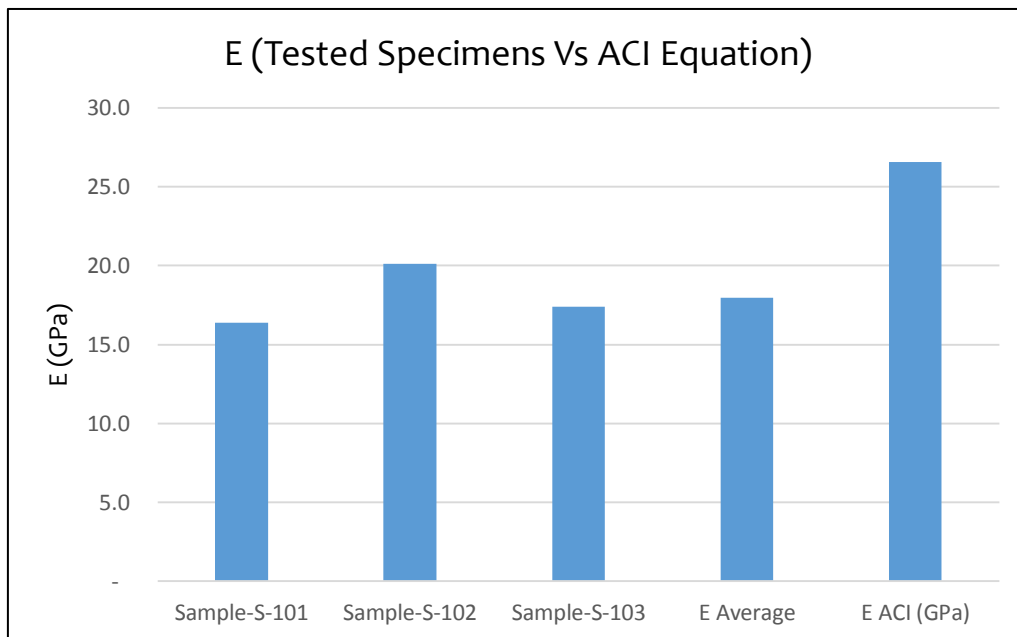


Figure 4.6: Tested Vs ACI Calculated Modulus of Elasticity

4.3: MORPHOLOGY AND PORE STRUCTURE

Five electron images were captured allowing to investigate the specimen morphology deeper and get a close view of the microstructure of such new concrete type.

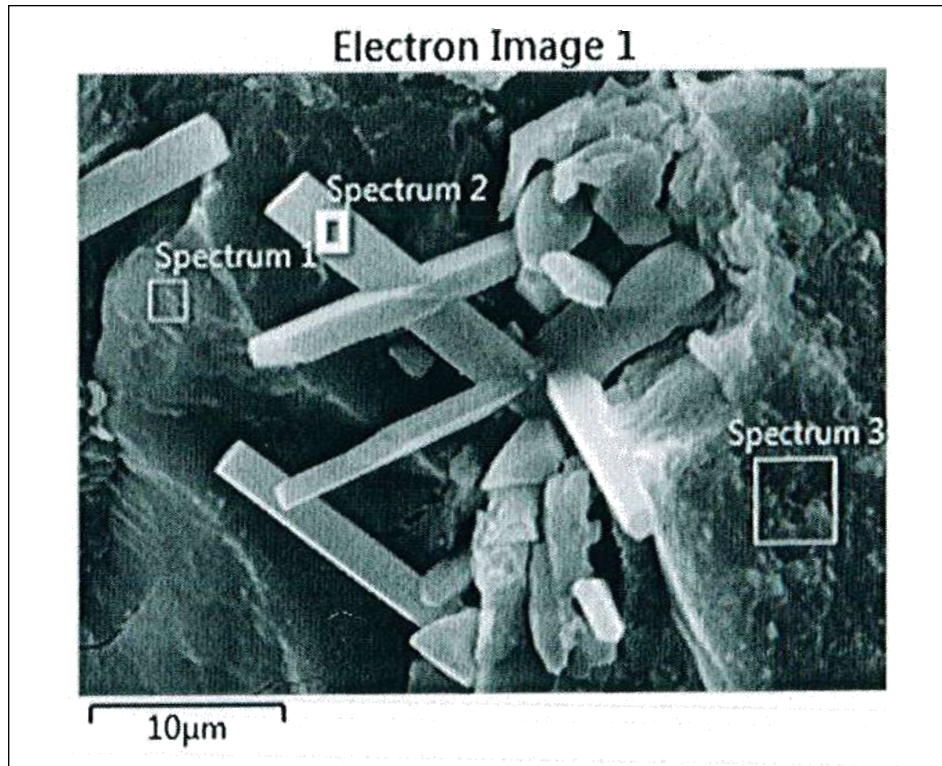


Figure 4.7: Electron Image-1

Figure 4.7 shows electron image-1 for specimen's surface particles to the scale of 10 micron. Due to the inconsistency of the grain shapes and sizes, three energy spectra were generated for three different locations at the electron image. In some part of the electron image, the material compositions form a homogeneous paste (areas where spectrum 1, and spectrum 3 were generated for) while particles were left free of bond in other areas of the investigated specimen surface (i.e. where spectrum 2 was generated for).

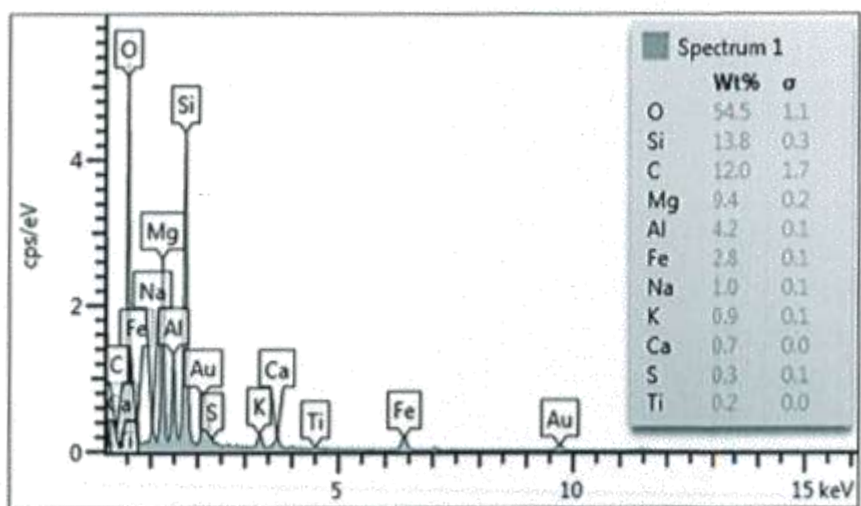


Figure 4.8: EDS Spectrum-1

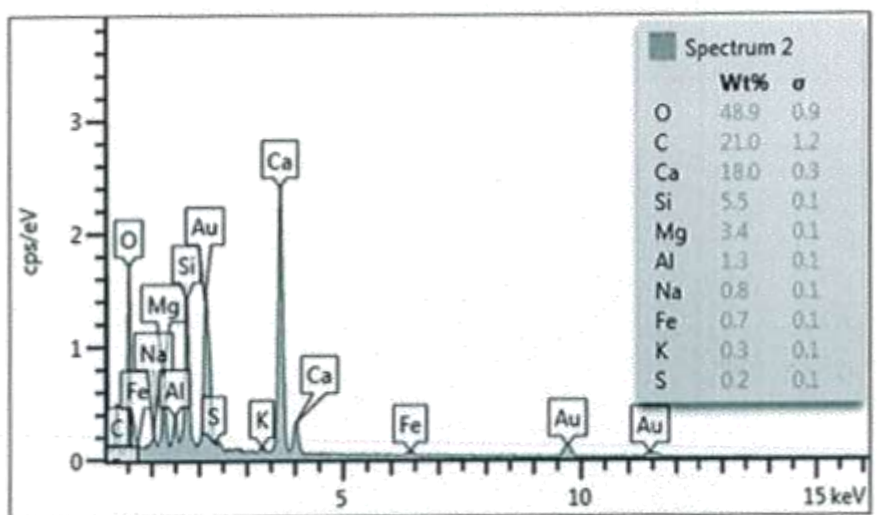


Figure 4.9: EDS Spectrum-2

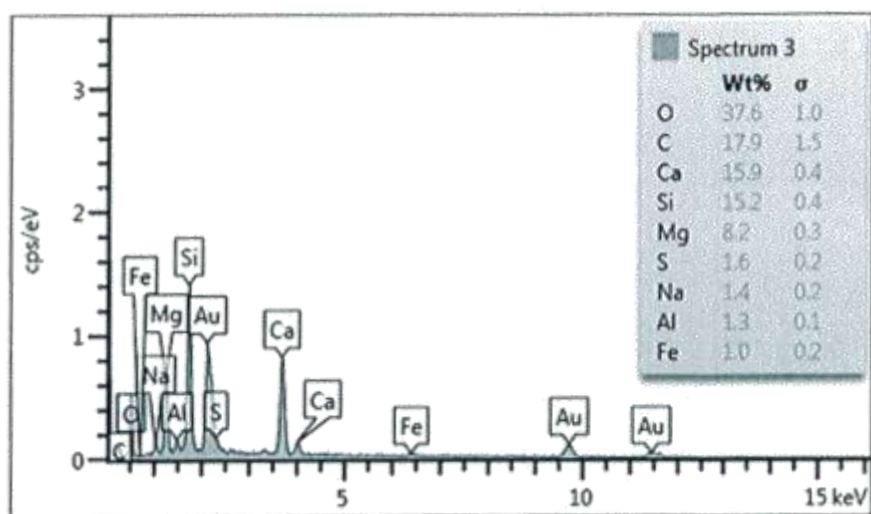


Figure 4.10: EDS Spectrum-3

The previous three Figures (4.8, 4.9, and 4.10) plot the ED spectra taken at three different location in electron image-1. In all spectra, **O** and **C** were major components present in the sample. In spectrum-1 and spectrum-3, **Si** and **Mg** were available with higher percentage than in spectrum-2. This might indicate that in these areas of the sample, the hydration was more complete than the area where spectrum-2 was generated. **Ca** was present in a high percentage in spectrum-2 and spectrum-3 unlike spectrum-1. Other elements were trace elements with less than 1%. It is important to note that Au peaks which were detected in all spectra were resulted from gold sputtering process and it was not an element of our sample being tested.

In the next Figure 4.11, electron image-2 was produced for specimen's surface particles to the scale of 5 micron. In the upper part of the image, a line of around 0.1 micron was detected. This might indicates a crack at the surface where the image was captured. As illustrated in electron image-2, the majority of the particles were similar in shape even though they do vary in size. One spectrum was generated from this image as shown in the same Figure.

Figure 4.12 plots the ED spectrum-4. In this spectrum, **O**, **Ca**, and **C** were major components present in the sample with lesser percentage of **Si** and **Mg**. Other elements were minor ones.

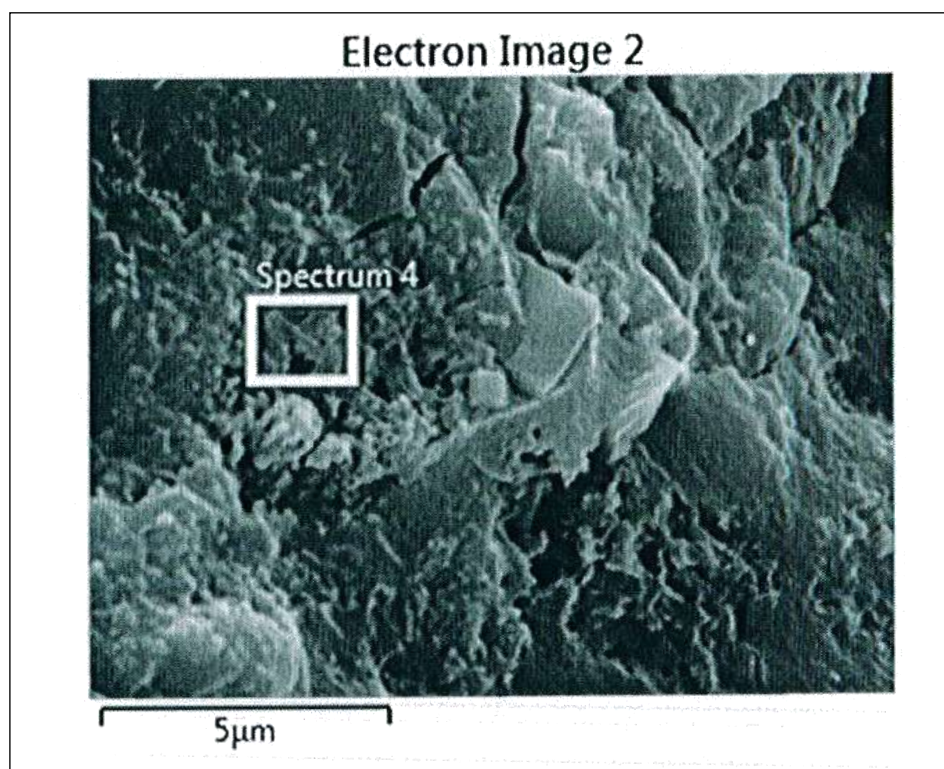


Figure 4.11: Electron Image-2

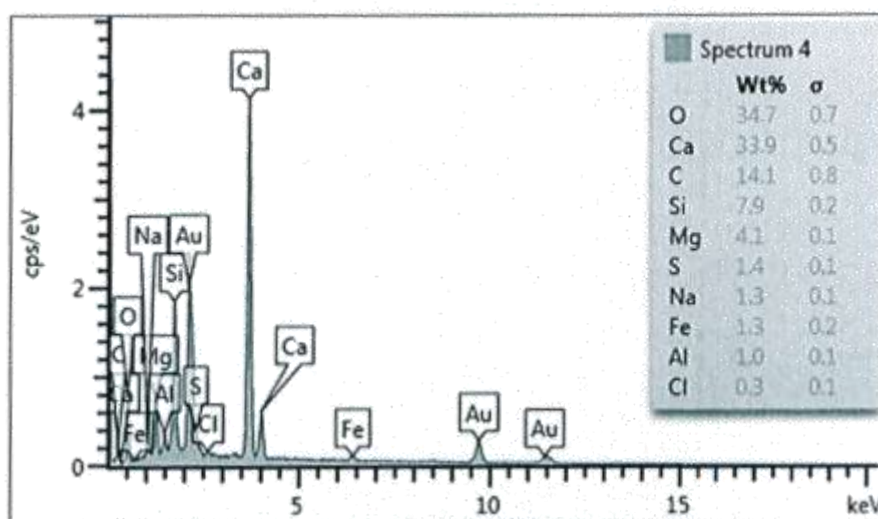


Figure 4.12: EDS Spectrum-4

Another electron image-(#3) was captured with specimen's surface particles to the scale of 50 micron as presented in Figure 4.13. Two spectra (5 and 6) were generated from this image-3 as shown in Figure 4.14, and 4.15 respectively.

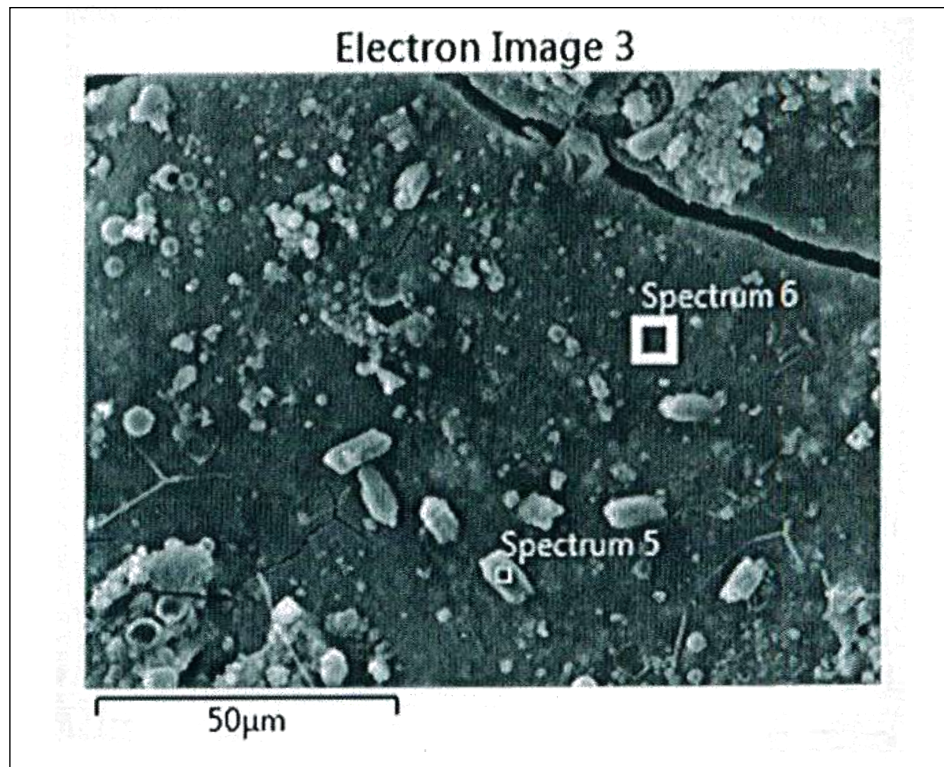


Figure 4.13: Electron Image-3

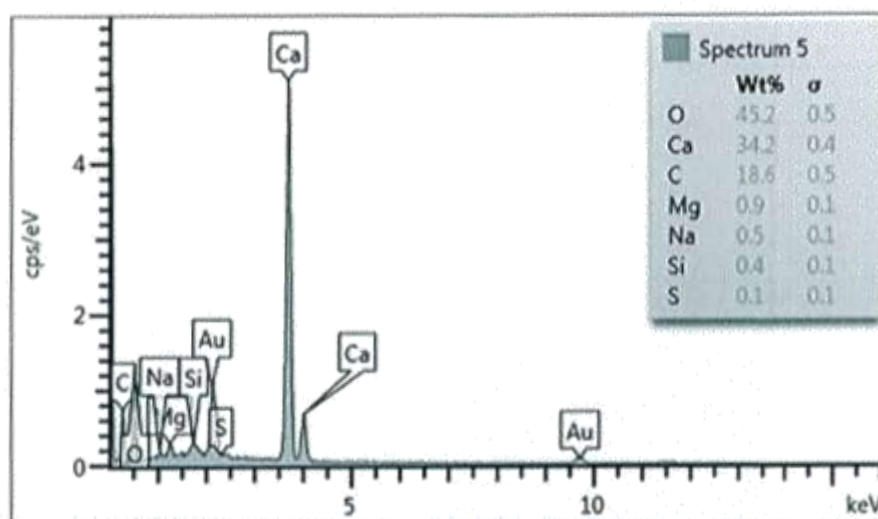


Figure 4.14: EDS Spectrum-5

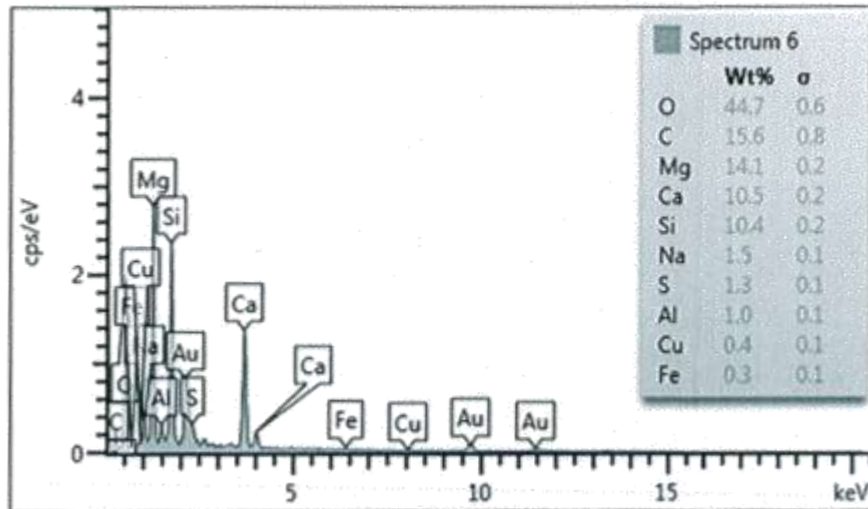


Figure 4.15: EDS Spectrum-6

Similarly, in Figures 4.16 through Figure 4.18, electron images 4 and 5 were captured and corresponding spectra 7 and 8 for those images were generated respectively as shown in Figure 4.17 and Figure 4.19. For these two electron images, the energy spectra were generated for a larger area of the electron image. A wider area was investigated.

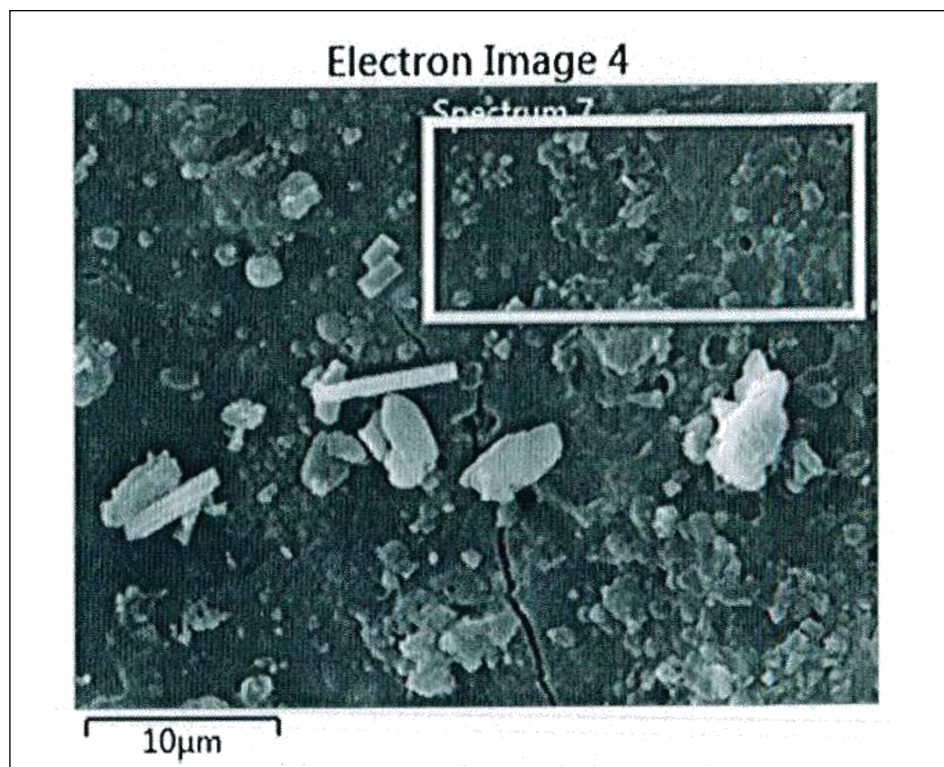


Figure 4.16: Electron Image-4

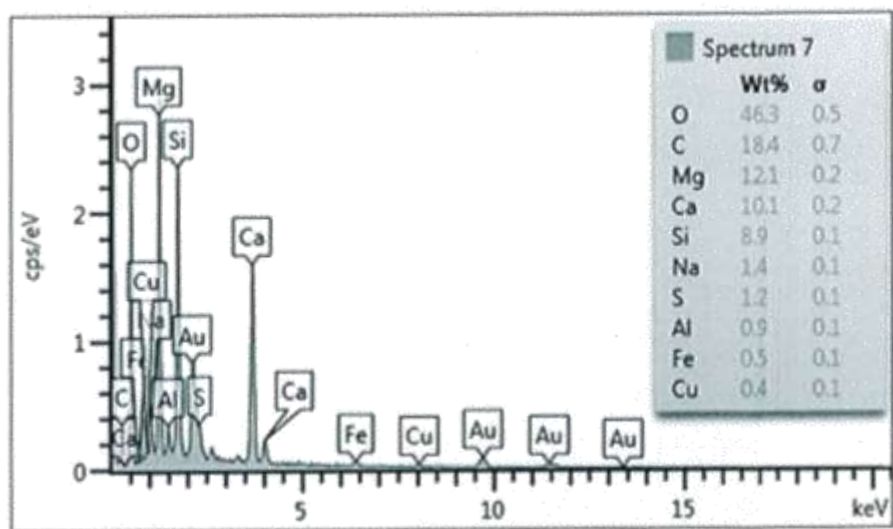


Figure 4.17: EDS Spectrum-7

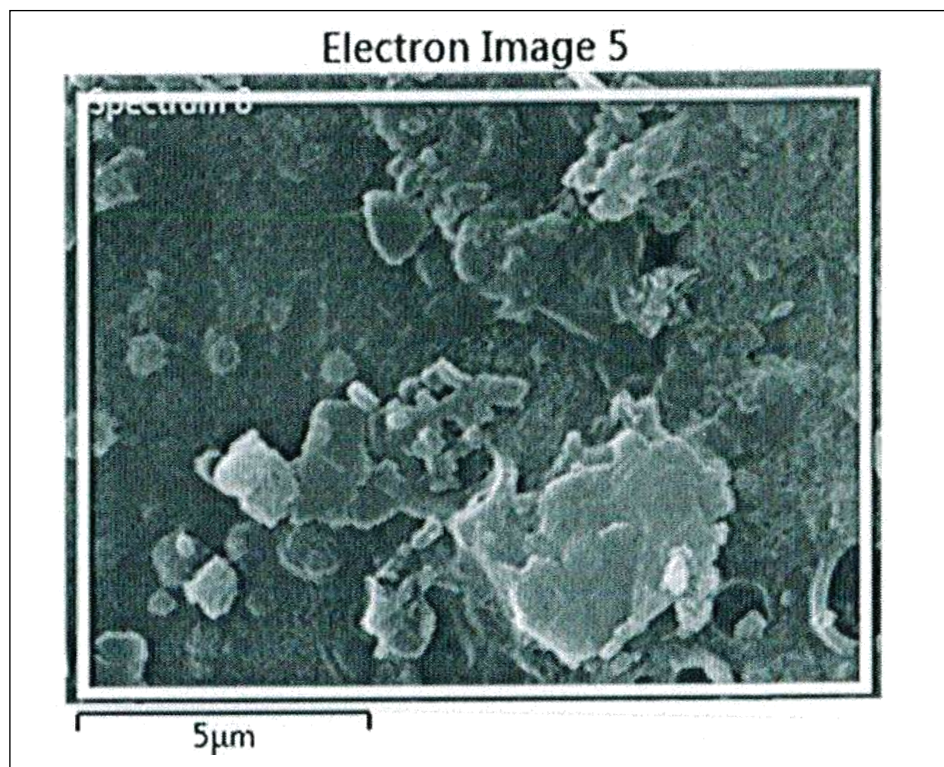


Figure 4.18: Electron Image-5

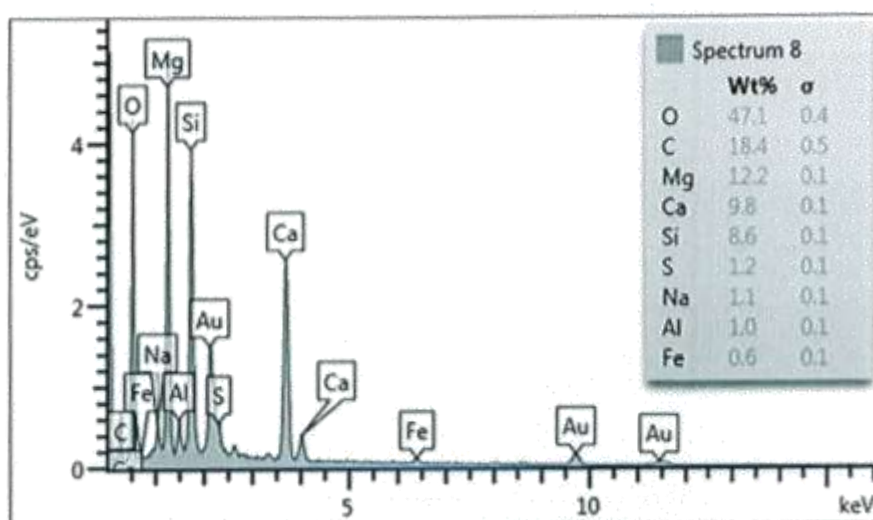


Figure 4.19: EDS Spectrum-8

4.4: DURABILITY

The ability to withstand the intended exposure conditions is an important durability measure of concrete. Reinforcement corrosion was investigated as well as sulfate attack.

Reinforcement Corrosion:

Both potential difference and corrosion current density were measured as the time passes. Potential differences in (mV) data measured for the tree samples (S-71, S-72, and S73) were tabulated in Table 4.8. Potential difference was plotted vs time in days as presented in Figure 4.20.

Table 4.8: Corrosion Potentials Results

Specimen (S-71)		Specimen (S-72)		Specimen (S-73)	
Time (Days)	Corrosion potential (mV, SCE)	Time (Days)	Corrosion potential (mV, SCE)	Time (Days)	Corrosion potential (mV, SCE)
14	-184	14	-165	14	-186
30	-284	30	-246	30	-286
45	-440	45	-384	45	-463
60	-610	60	-554	60	-633
90	-633	90	-612	90	-647
120	-663	120	-618	120	-680
150	-662	150	-622	150	-678
180	-661	180	-638	180	-691
210	-661	210	-644	210	-709
240	-658	240	-647	240	-699
270	-668	270	-642	270	-694

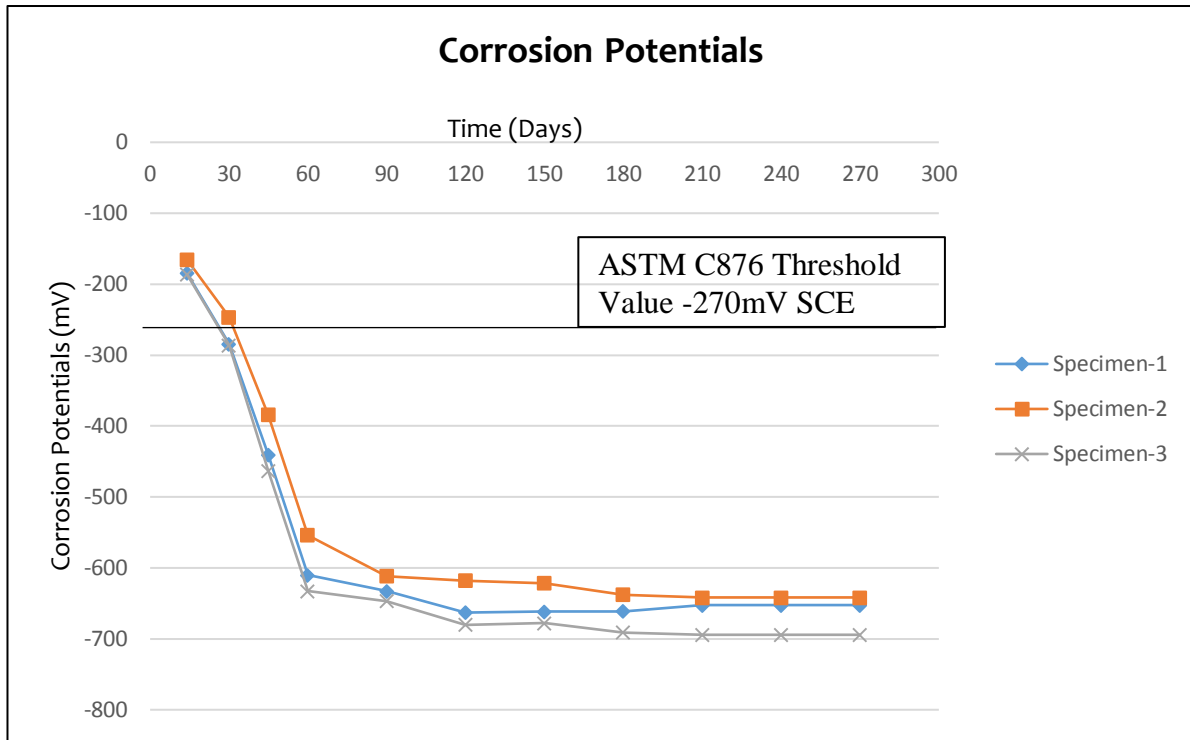


Figure 4.20: Corrosion Potentials Vs Time

In addition, Corrosion current density (I_{corr}) results on steel in the concrete specimens are tabulated in table 4.9 I_{corr} in ($\mu\text{A}/\text{cm}^2$) was plotted against time in days as seen in Figure 4.21.

Table 4.9: Corrosion Current Density Results

I_{corr} ($\mu\text{A}/\text{cm}^2$)			
Time (Days)	S-71	S-72	S-73
14	0.161	0.146	0.171
30	0.166	0.156	0.176
45	0.176	0.166	0.186
60	0.181	0.169	0.191
90	0.197	0.187	0.209
120	0.239	0.227	0.256
150	0.271	0.263	0.292
180	0.315	0.303	0.336
210	0.347	0.335	0.368
240	0.367	0.355	0.388
270	0.413	0.398	0.428

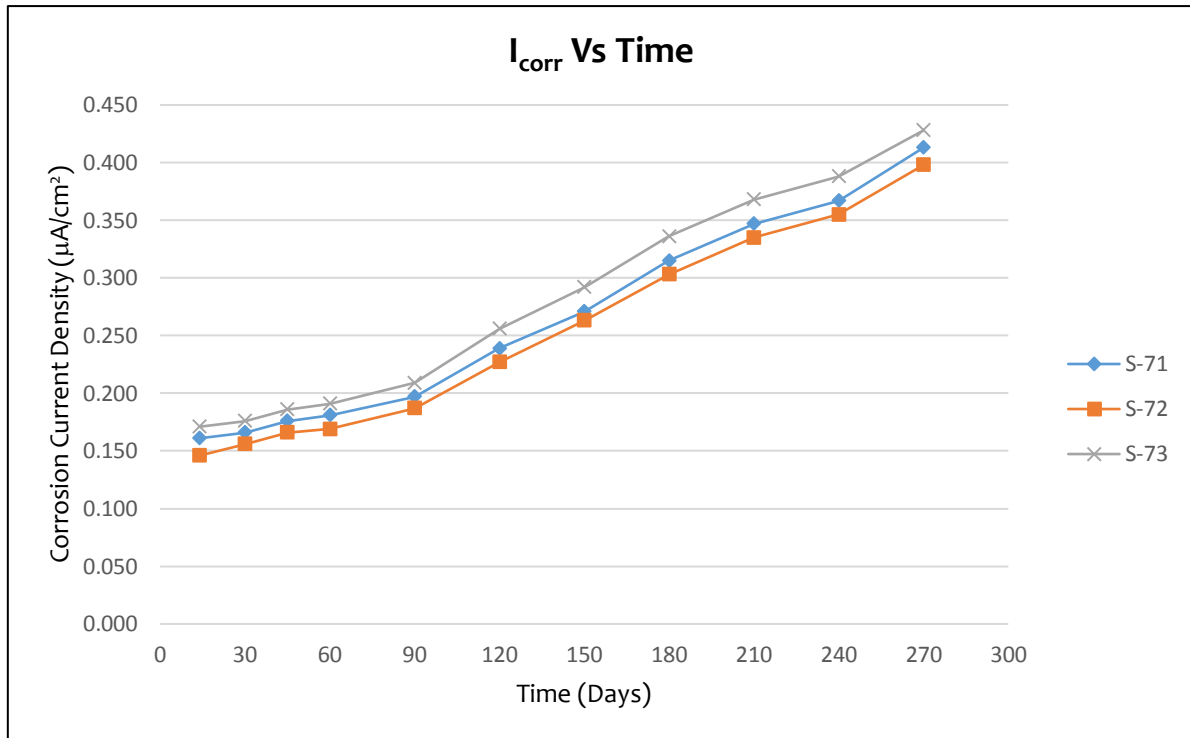


Figure 4.21: Corrosion Current Density Vs Time

Sulfate Attack:

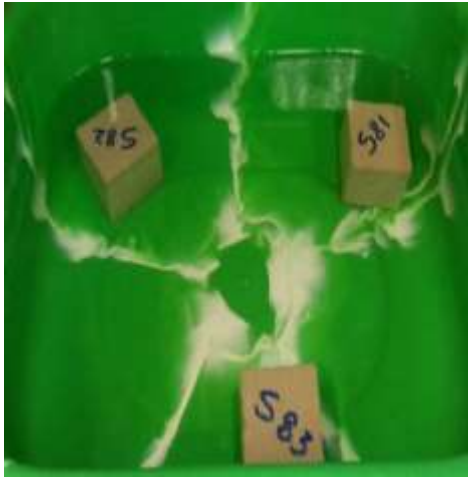
Visual Examination

Thorough visual examination was carried out for 90-days, 180-days, and 270-days exposure of the specimens to (NaSO₄ + MgSO₄) solution. Signs of Spalling, edge softening, and cracking were evaluated visually.

Visual examination for 90-days as well as 180-days of exposure showed that the specimens were in a very good conditions, this means that there was no visual evidence of severe spalling on the specimens' surface.

For exposure time of 270-days, still specimens under examination were found in good conditions with little softening at the corners. Also, a little amount of a yellow powder was found in the (NaSO₄ + MgSO₄) solution – most likely this powder is the fine

materials covering the specimens' edges which were affected by solution that were emerged in.



After 90 days



After 270 days

Weight Loss

Weights of exposed Specimens to ($\text{NaSO}_4 + \text{MgSO}_4$) solution were monitored for any anticipated loss due to the attack of sulfate. Weight of specimens were measured after 90-days, 180-days, and 270-days of exposure to sulfate solution. Table 4.10 shows weight loss due to sulfate attack. In addition, as exposure time increases, the specimens loses more and more weight. Moreover, this loss was expected as some yellow powder was visually detected in the sulfate solution in which the specimens were submerged.

Table 4.10: Weight Loss Due to Sulfat attack

Exposure (days)	Specimen #	Weight (g)	Weight Loss %	Avg. Weight Loss %
0	Spec-1	260.1	0.00%	0.00%
	Spec-2	261.0	0.00%	
	Spec-3	260.5	0.00%	
90	Spec-1	259.8	0.12%	0.12%
	Spec-2	260.7	0.11%	
	Spec-3	260.2	0.12%	
180	Spec-1	259.2	0.35%	0.38%
	Spec-2	260.0	0.38%	
	Spec-3	259.4	0.42%	
270	Spec-1	257.3	1.08%	1.14%
	Spec-2	258.4	1.00%	
	Spec-3	257.0	1.34%	

CHAPTER 5

CONCLUSIONS AND RECOMMENDATIONS

5.1: CONCLUSIONS

Following conclusions are made based on the data developed in this study:

- ❖ Three potential alkali-activated binding materials were investigated for full replacement of Portland cement, i.e. Cement Kiln Dust (CKD), Limestone Powder (LSP), and Baghouse Dust (BHD).
- ❖ According to this study, only one of the three investigated industrial waste materials, i.e. CKD was proven to be a successful binding material that can replace Portland cement and result in a sound concrete mix with reasonable properties.
- ❖ LSP and BHD were not investigated further than the trial mixes stage. This was because all mixes that were prepared incorporating each of them failed to achieve a reasonable compressive strength. The maximum compressive strengths achieved by trial mixes prepared using LSP and BHD were as low as 6.7 MPa and 9.0 MPa respectively which is an extremely low value to be accepted as concrete mix.

- ❖ Several mechanical properties were measured for the developed CKD-based new geopolymer concrete type and very reasonable results were obtained and then compared to values that can be calculated theoretically and recommended by ACI-318 Code for structural concrete.
- ❖ The compressive strength (f'_c) was as high as (25 MPa). This is a reasonable to be used as structural concrete.
- ❖ The modulus of rupture (f_r) was measured to be (2.97 MPa) for the same mix that achieved 25 MPa compressive strength. Comparing to the value calculated of modulus of rupture, using ACI-318 code with $f'_c = 25$ MPa, which was resulted to be almost 23 MPa shows that the difference is very small which indicates that the new developed geopolymer concrete using CKD-based alkali-activated binder might possess similar properties of conventional concrete prepared using ordinary Portland cement.
- ❖ A relatively lower modulus of elasticity (E) measured to be (18.0 GPa) compared to the value that can be calculated to be (26.5 GPa) using the equation provided by ACI-318 code.
- ❖ Almost all Energy Dispersive Spectroscopy spectra generated using the Scanning Electron Microscopy images captured indicate that **O** and **C** were major components present in the samples with more than 10% while **Mg**, **Ca**, and **Si** were minor ones with other elements like Al, Fe, Na, S, and other materials present as trace elements with less than 1%.

- ❖ Corrosion measurements (according to ASTM standard C876) may NOT be valid for this type of cement. Further investigation to develop a criteria for alkaline-activated binders is required.
- ❖ However, very good sulfate resistance was indicated by very low loss of weight after 270 days of exposure to (NaSO₄ + MgSO₄) solution.

5.2: RECOMMENDATIONS

Recommendations of optimum ratio of (Na₂SiO₃/NaOH) as well as the application of the developed alkali-activated concrete using CKD, LSP, and BHD is summarized in Table 5.1.

Table 5.1: Recommendations summary

Material	Strength	Optimum (Na ₂ SiO ₃ /NaOH)	Application
CKD	> 20 MPa	2.5	structural concrete
LSP	< 10 MPa		non-structural concrete
BHD			

5.3: FUTURE WORK

Based on this study results and conclusions drawn earlier, the following are the recommendations for future research:

- ❖ More investigations to be carried out to improve the properties of concrete mixes prepared utilizing CKD as the main binding cementous material.

- ❖ Deeper investigations of the microstructure of such new binder is highly recommended in order to provide more information on pore structure, thus leading to better properties and concrete quality.
- ❖ Further research is required to develop the workability of such binder. This step will help to bring this binder to the real practice.
- ❖ Further research is required to study, investigate and improve the durability of such type of concrete.

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APPENDICES

APPENDIX-A



Chemical Marketing & Distribution Co. Ltd.

Material Safety Data Sheets SODIUM SILICATE

1 Product Information and Company Identification

Chemical	Sodium Silicate
Synonyms	Water Glass; Soluble Glass; Silicate of Soda; Egg Preserver
Chemical Formula	$\text{Na}_2\text{O}(\text{SiO}_2)_x \cdot (\text{H}_2\text{O})_x$
CAS No	Not applicable to mixtures.
Molecular Weight	Not applicable to mixtures
Product Number/s	
Supplier	Chemical Marketing and Distribution Co. Ltd. (CMDC)
Address	P. O. Box 1053 Dammam 31431 Saudi Arabia
Telephone Number	966 03 8472466
Facsimile Number	966 03 8472648
E Mail Address	sales@cmdc.com.sa
Manufacturer	Basic Chemical Industries Ltd. (BCI), Saudi Arabia

2 Composition/Information on Ingredients

<u>Ingredients</u>	<u>CAS No</u>	<u>Percent</u>	<u>Hazardous</u>
Sodium Silicate	1344-09-8	35 - 40%	Yes
Water	7732-18-5	60 - 65%	No

3 Hazards Identification

Emergency Overview	Warning! Harmful if swallowed or inhaled. Causes severe irritation to eyes, skin and respiratory tract.
Potential Health Effects	Diluted solutions of sodium silicate are strong alkaline irritants. The solid sodium silicate is corrosive. Exposure to alkaline corrosives may result in severe burns depending on the concentration and duration of exposure. Sodium silicate is a type of amorphous silica and does not cause pulmonary silicosis.

Inhalation	A strong alkaline irritant. Inhalation can cause severe irritation of mucous membranes and upper respiratory tract. Symptoms may include burning sensation, coughing, wheezing, laryngitis, and shortness of breath, headache, and nausea and vomiting. High concentrations may cause lung damage.
Ingestion	A strong alkaline irritant. Causes irritation to the gastrointestinal tract. Symptoms may include nausea, vomiting and diarrhea. Solid sodium silicate: Alkaline corrosive ingestion may produce burns to the lips, tongue, oral mucosa, upper airway, and esophagus and occasionally stomach.
Skin Contact	A strong alkaline irritant. Causes severe irritation. Symptoms include redness, itching and pain. Dries to form a glass film, which can cut skin. Solid sodium silicate: Dermal contact with alkaline corrosives may produce pain, redness, severe irritation or full thickness burns.
Eye Contact	A strong alkaline irritant. Alkaline eye exposures produce severe irritation with effects similar to those of dilute caustics. Inflammation or burns with possible damage to the eye tissues can occur together with tearing and considerable pain.
Chronic Exposure	No information found.
Aggravation of Pre-existing Condition	Persons with pre-existing skin disorders or impaired respiratory function may be more susceptible to the effects of the substance.

4 First Aid Measures

Inhalation	If inhaled, remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. Get medical attention if irritation persists.
Ingestion	Give several glasses of water to drink to dilute. If large amounts were swallowed, get medical advice.
Skin Contact	Not expected to require first aid measures. Wash exposed area with soap and water. Get medical advice if irritation develops.
Eye Contact	Wash thoroughly with running water. Get medical advice if irritation develops.
Note To Physician	

5 Fire Fighting Measures

Fire	Not considered being a fire hazard.
Explosion	Not considered being an explosion hazard.
Fire Extinguishing Media	Use any means suitable for extinguishing surrounding fire.
Special Information	In the event of a fire, wear full protective clothing and NIOSH-approved self-contained breathing apparatus with full facepiece operated in the pressure demand or other positive pressure mode.

6 Accidental Release Measures

Caution! Floor and other surfaces may be slippery. Ventilate area of leak or spill. Wear appropriate personal protective equipment as specified in Section 8. Contain and recover liquid when possible. Collect liquid in an appropriate container or absorb with an inert material (e. g., vermiculite, dry sand, and earth), and place in a chemical waste container. Do not use combustible materials, such as sawdust. Do not flush to sewer!

7 Handling And Storage

Keep in a tightly closed container, stored in a cool, dry, ventilated area. Protect against physical damage. Isolate from incompatible substances. Containers of this material may be hazardous when empty since they retain product residues (vapors, liquid); observe all warnings and precautions listed for the product.

8 Exposure Controls/Personal Protection

Airborne Exposure Limits	<p>Amorphous Silica, including natural diatomaceous earth:</p> <ul style="list-style-type: none"> - OSHA Permissible Exposure Limit (PEL): (80 mg/m³) / (%SiO₂) (TWA). - ACGIH Threshold Limit Value (TLV): 10 mg/m³ (TWA) inhalable particulate; 3 mg/m³ (TWA) respirable particulate.
Ventilation System	<p>A system of local and/or general exhaust is recommended to keep employee exposures below the Airborne Exposure Limits. Local exhaust ventilation is generally preferred because it can control the emissions of the contaminant at its source, preventing dispersion of it into the general work area.</p> <p>Please refer to the ACGIH document, Industrial Ventilation, A Manual of Recommended Practices, most recent edition, for details.</p>
Personal Respirators (NIOSH Approved)	<p>If the exposure limit is exceeded, a half-face dust/mist respirator may be worn for up to ten times the exposure limit or the maximum use concentration specified by the appropriate regulatory agency or respirator supplier, whichever is lowest.</p> <p>A full-face piece dust/mist respirator may be worn up to 50 times the exposure limit, or the maximum use concentration specified by the appropriate regulatory agency, or respirator supplier, whichever is lowest.</p> <p>For emergencies or instances where the exposure levels are not known, use a full-facepiece positive-pressure, air-supplied respirator.</p> <p>WARNING: Air-purifying respirators do not protect workers in oxygen-deficient atmospheres.</p>
Skin Protection	<p>Wear impervious protective clothing, including boots, gloves, lab coat, apron or coveralls, as appropriate, to prevent skin contact.</p>
Eye Protection	<p>Use chemical safety goggles and/or a full face shield where splashing is possible. Maintain eye wash fountain and quick-drench facilities in work area.</p>

9 Physical And Chemical Properties

Appearance	Clear to cloudy, viscous liquid.
Odor	Odorless.
Solubility	Complete (100%).
Specific Gravity	1.3 - 1.5.
pH	11 - 12.5.
Boiling Point	102C (216F).
Melting Point	No information found.
Vapor Density (Air=1)	Not applicable.
Vapor Pressure (mm Hg)	18 @ 20C (68F).
Evaporation Rate (BuAc=1)	No information found.
% Volatiles by volume @ 21C (70F)	0

10 Stability And Reactivity

Stability	Stable under ordinary conditions of use and storage.
Hazardous Decomposition Products	No information found.
Hazardous Polymerization	Will not occur.
Incompatibilities	Fluorine, mineral acids, organic acids, organic materials. May produce hydrogen gas on prolonged contact with metals. Gels when mixed with acids.
Conditions to Avoid	Incompatibles.

11 Toxicological Information

No LD50/LC50 information found relating to normal routes of occupational exposure.

-----\Cancer Lists-----

---NTP Carcinogen---

Ingredient Known Anticipated IARC Category

Sodium Silicate (1344-09-8) No None

Water (7732-18-5) No None

12 Ecological Information

Environmental Fate	No information found.
Environmental Toxicity	No information found.

13 Disposal Considerations

Whatever cannot be saved for recovery or recycling should be managed in an appropriate and approved waste disposal facility. Processing, use or contamination of this product may change the waste management options. State and local disposal regulations may differ from federal disposal regulations. Dispose of container and unused contents in accordance with federal, state and local requirements.

14 Transport Information

Not regulated

15 Regulatory Information

16 Other Information

NFPA Ratings	Health: 2 Flammability: 0 Reactivity: 0 .
Label Hazard Warning	Warning! Harmful if swallowed or inhaled. Causes severe irritation to eyes, skin and respiratory tract.
Label Precautions	Avoid contact with eyes, skin and clothing. Wash thoroughly after handling. Avoid breathing vapor or mist. Keep container closed. Use only with adequate ventilation.
Label First Aid	If swallowed, DO NOT INDUCE VOMITING. Give large quantities of water. Never give anything by mouth to an unconscious person. If inhaled, remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. In case of contact, immediately flush eyes or skin with plenty of water for at least 15 minutes. Remove contaminated clothing and shoes. Wash clothing before reuse. In all cases, get medical attention. Health Rating: 1 - Slight Flammability Rating: 0 - None Reactivity Rating: 0 - None Contact Rating: 2 – Moderate.

APPENDIX-B

Construction

Product Data Sheet
Edition 10/07/2010
Identification no:
02 13 01 01 100 0 000678
Sika® ViscoCrete®-10 R

Sika® ViscoCrete®-10 R

High performance superplasticiser cum retarder

Product Description	Sika® ViscoCrete®-10 R is a third generation superplasticizer for concrete and mortar. It meets the requirements for superplasticizers according IS 9103 – 1999 (Amended 2003).
Uses	<p>Sika® ViscoCrete®-10R is suitable for the production of precast concrete. Sika® ViscoCrete®-10R facilitates extreme water reduction, excellent flowability with the same time optimal cohesion and highest self compacting behaviour. Sika® ViscoCrete®-10R is used for the following types of concrete.</p> <ul style="list-style-type: none"> ■ Precast concrete ■ Self compacting ■ Concrete with highest water reduction (up to 30%) ■ High strength concrete with prolonged slump retention ■ High water reduction, excellent flowability, coupled with high early strengths, have a positive influence on the above mentioned applications.
Characteristics / Advantages	<p>Sika® ViscoCrete®-10R acts by different mechanisms. Through surface adsorption and sterical separation effect on the cement particles, in parallel to the hydration process, the following properties are obtained:</p> <ul style="list-style-type: none"> ■ Strong self compacting behaviour, therefore suitable for the production of self compacting concrete (S.C.C) ■ Extremely high water reduction (resulting in high density and strengths) ■ Excellent flowability (resulting in highly reduced placing and compacting efforts) ■ Improved shrinkage and creep behaviour ■ Reduced rate of carbonation of the concrete ■ It does not contain chloride or other steel corrosion promoting ingredients. It may therefore be used without any restrictions for reinforced- and prestressed – concrete construction.
Tests	
Approval / Standards	IS 9103 – 1999 (Amended 2003).
Product Data	
Form	
Appearance / Colour	Brown liquid
Packaging	100 kg, 200 kg
Storage	
Storage Conditions / Shelf -Life	12 months from date of production if stored properly in undamaged unopened, original sealed packaging, in dry conditions at temperatures between +5 °C and +30 °C. Protect from direct sunlight and frost.



1
Sika® ViscoCrete®-10 R 1/3

Technical Data	
Chemical Base	Aqueous solution of modified polycarboxylate
Relative Density	1.08 kg/l at 30°C
System Information	
Application Details	
Consumption / Dosage	<ul style="list-style-type: none"> ■ For soft plastic concrete 0.2 – 0.6% by weight of cement ■ For flowing self compacting concrete (S.C.C) 0.6 – 2.0% by weight of cement Actual dosage to be finalised on the basis of site trials.
Application Conditions / Limitations	
Compatibility	Sika® ViscoCrete®-SC 001 may be combined with many other Sika® Products. Important: Always conduct trials before combining products in specific mixes and contact our Technical Service Department for information about specific combinations.
Application Instructions	
Dispensing	Sika® ViscoCrete®-10R is added to the gauging water or simultaneously with it poured into the concrete mixer. For optimum utilization of the high water reduction we recommend thorough mixing at a minimal wet mixing time of 60 seconds. The addition of the remaining gauging water to fine tune the concrete consistency may only be started after 2/3 minutes of the wet mixing time, to avoid surplus water in the concrete.
Application Method / Tools	With the use of Sika® ViscoCrete®-10R concrete of highest quality is being produced. The standard rules of good concreting practice (production as well as placing) must also be observed with Sika® ViscoCrete®-10R concrete. Fresh concrete must be cured properly.
Cleaning of Tools	Clean all tools and application equipment with water immediately after use. Hardened / cured material can only be mechanically removed.
Notes on Application / Limitations	If frozen and / or if precipitation has occurred, Sika® ViscoCrete®-10R may be used after thawing slowly at room temperature and after intensive mixing.
Value Base	All technical data stated in this Product Data Sheet are based on laboratory tests. Actual measured data may vary due to circumstances beyond our control.
Health and Safety Information	For information and advice on the safe handling, storage and disposal of chemical products, users shall refer to the most recent Material Safety Data Sheet containing physical, ecological, toxicological and other safety-related data.

Legal Notes

The information, and, in particular, the recommendations relating to the application and end-use of Sika products, are given in good faith based on Sika's current knowledge and experience of the products when properly stored, handled and applied under normal conditions in accordance with Sika's recommendations. In practice, the differences in materials, substrates and actual site conditions are such that no warranty in respect of merchantability or of fitness for a particular purpose, nor any liability arising out of any legal relationship whatsoever, can be inferred either from this information, or from any written recommendations, or from any other advice offered. The user of the product must test the product's suitability for the intended application and purpose. Sika reserves the right to change the properties of its products. The proprietary rights of third parties must be observed. All orders are accepted subject to our current terms of sale and delivery. Users must always refer to the most recent issue of the local Product Data Sheet for the product concerned, copies of which will be supplied on request.



Sika India Pvt. Ltd.
Commercial Complex II
820, Diamond Harbour Road
Kolkata, 700 034, India

Phone +91 33 2447 2448/2449
Telefax +91 33 2396 8688
ind.sika.com
info.india@in.sika.com

APPENDIX-C

Specimen-1			Specimen-2			Specimen-3		
Extension (mm)	Load (N)	Flexural Strength (Mpa)	Extension (mm)	Load (N)	Flexural Strength (Mpa)	Extension (mm)	Load (N)	Flexural Strength (Mpa)
0.000	5	0.020	0.035	8	0.030	0.000	13	0.049
0.000	12	0.043	0.040	3	0.011	0.006	14	0.052
0.006	18	0.067	0.046	60	0.225	0.011	15	0.056
0.017	32	0.121	0.052	72	0.271	0.017	16	0.059
0.023	47	0.178	0.057	85	0.317	0.029	14	0.052
0.029	58	0.218	0.067	77	0.287	0.035	44	0.165
0.034	69	0.258	0.075	93	0.348	0.040	48	0.180
0.046	79	0.298	0.080	109	0.409	0.052	60	0.224
0.052	90	0.338	0.086	119	0.445	0.057	85	0.319
0.057	112	0.419	0.092	122	0.459	0.063	122	0.458
0.069	120	0.450	0.098	126	0.472	0.075	174	0.651
0.075	127	0.476	0.104	129	0.485	0.080	174	0.653
0.080	136	0.510	0.115	131	0.490	0.086	175	0.655
0.086	143	0.538	0.121	132	0.495	0.098	175	0.656
0.098	144	0.540	0.126	133	0.500	0.104	176	0.658
0.103	145	0.542	0.132	135	0.504	0.109	176	0.660
0.109	145	0.544	0.138	136	0.509	0.121	177	0.662
0.115	146	0.547	0.144	137	0.512	0.126	177	0.664
0.126	146	0.549	0.149	137	0.513	0.138	177	0.666
0.132	147	0.551	0.161	137	0.514	0.144	178	0.667
0.138	148	0.553	0.167	137	0.515	0.149	178	0.669
0.149	148	0.556	0.172	138	0.516	0.161	179	0.671
0.155	149	0.558	0.178	138	0.517	0.167	179	0.673
0.161	136	0.511	0.184	138	0.518	0.172	180	0.675
0.167	142	0.532	0.190	145	0.545	0.184	180	0.676
0.178	147	0.552	0.195	161	0.603	0.190	168	0.632
0.184	153	0.573	0.207	157	0.588	0.195	160	0.599
0.190	163	0.612	0.213	158	0.592	0.207	160	0.600
0.195	166	0.622	0.218	159	0.597	0.213	160	0.600
0.207	168	0.631	0.224	161	0.603	0.224	160	0.600
0.213	171	0.641	0.230	168	0.630	0.230	160	0.601
0.218	173	0.650	0.236	146	0.549	0.236	160	0.601
0.224	176	0.660	0.247	146	0.548	0.247	160	0.601
0.236	149	0.560	0.253	146	0.548	0.253	171	0.641
0.241	149	0.559	0.259	146	0.547	0.259	175	0.658
0.247	149	0.558	0.264	146	0.547	0.270	176	0.659
0.259	149	0.558	0.270	146	0.546	0.276	198	0.741
0.264	150	0.563	0.276	146	0.546	0.282	208	0.779
0.270	151	0.567	0.282	145	0.546	0.293	218	0.817
0.276	153	0.572	0.293	147	0.552	0.299	243	0.911
0.287	154	0.577	0.299	168	0.632	0.305	269	1.010
0.293	161	0.604	0.305	189	0.709	0.316	279	1.047
0.299	178	0.669	0.310	210	0.786	0.322	286	1.074
0.305	196	0.733	0.316	230	0.863	0.333	300	1.124
0.316	213	0.798	0.322	251	0.941	0.339	318	1.191
0.322	230	0.861	0.328	273	1.024	0.345	343	1.286
0.328	245	0.920	0.339	296	1.109	0.356	368	1.381
0.333	261	0.978	0.345	318	1.193	0.362	394	1.476
0.345	276	1.036	0.351	341	1.277	0.368	419	1.571
0.350	292	1.094	0.356	363	1.362	0.379	444	1.665
0.356	307	1.153	0.362	386	1.446	0.385	468	1.755

0.368	324	1.214	0.368	408	1.530	0.391	492	1.845
0.373	345	1.292	0.374	431	1.615	0.402	516	1.935
0.379	366	1.371	0.385	453	1.699	0.408	540	2.025
0.385	387	1.450	0.391	481	1.805	0.414	564	2.116
0.396	408	1.528	0.397	513	1.924	0.425	588	2.206
0.402	460	1.726	0.402	545	2.042	0.431	612	2.296
0.408	482	1.809	0.408	576	2.161	0.443	650	2.437
0.414	504	1.891	0.414	617	2.312	0.448	670	2.513
0.425	526	1.974	0.425	660	2.476	0.454	662	2.483
0.431	548	2.056	0.431	692	2.593	0.465	693	2.598
0.437	570	2.139	0.437	723	2.711	0.471	725	2.719
0.448	592	2.220	0.443	759	2.846	0.477	747	2.802
0.454	625	2.344	0.448	796	2.984	0.489	749	2.810
0.460	637	2.389	0.454	833	3.122	0.494	752	2.819
0.465	617	2.314	0.460	843	3.161	0.500	693	2.600
0.477	642	2.407	0.471	849	3.183	0.511	663	2.487
0.483	674	2.529	0.477	816	3.061	0.517	644	2.415
0.488	703	2.637	0.483	808	3.030	0.523	625	2.344
0.494	713	2.674	0.489	770	2.889	0.534	606	2.272
0.506	741	2.778	0.494	727	2.726	0.540	587	2.201
0.511	754	2.826	0.500	688	2.581	0.552	571	2.141
0.517	775	2.908	0.511	649	2.435	0.558	557	2.087
0.529	759	2.845	0.517	639	2.395	0.563	542	2.033
0.534	697	2.615	0.523	629	2.359	0.575	528	1.979
0.540	635	2.381	0.529	608	2.278	0.580	513	1.926
0.546	614	2.302	0.535	584	2.191	0.586	503	1.885
0.557	593	2.223	0.540	561	2.105	0.598	492	1.844
0.563	572	2.144	0.546	537	2.014	0.603	481	1.803
0.569	551	2.065	0.558	513	1.923	0.609	473	1.775
0.575	529	1.985	0.563	489	1.833	0.621	476	1.783
0.586	515	1.933	0.569	465	1.744	0.626	447	1.678
0.592	503	1.887	0.575	444	1.666	0.632	443	1.662
0.598	491	1.841	0.580	423	1.588	0.644	439	1.646
0.603	479	1.796	0.586	423	1.587	0.649	442	1.658
0.615	467	1.750	0.592	408	1.528	0.661	446	1.674
0.621	454	1.704	0.603	393	1.475	0.667	408	1.529
0.626	442	1.658	0.609	373	1.399	0.672	399	1.497
0.634	445	1.667	0.615	363	1.360	0.684	400	1.500
0.644	432	1.618	0.621	359	1.345	0.690	405	1.517
0.649	419	1.570	0.626	355	1.330	0.695	394	1.476
0.655	406	1.521	0.632	350	1.313	0.707	364	1.364
0.667	390	1.462	0.638	345	1.295	0.713	351	1.317
0.672	376	1.408				0.718	339	1.269
0.678	364	1.365				0.730	329	1.232
0.684	353	1.322				0.736	322	1.208
0.695	341	1.279				0.741	316	1.184
0.701	330	1.236				0.753	309	1.159
0.707	318	1.193				0.759	329	1.235

APPENDIX-D

Sample-S-103		
Load (KN)	Stress (Mpa)	Strain Gage-1 (10 ⁻⁶)
0	0.000	0
-2.5575	0.561	10
-4.115	0.902	19
-5.6725	1.244	29
-7.215	1.582	39
-8.505	1.865	49
-10.1875	2.234	60
-11.55	2.533	77
-12.6225	2.768	86
-13.985	3.067	100
-15.18	3.329	111
-16.8425	3.693	126
-18.06	3.960	142
-18.8325	4.130	153
-20.1375	4.416	169
-21.555	4.727	186
-23.145	5.075	204
-24.2975	5.328	219
-25.7375	5.644	241
-26.9975	5.920	261
-28.78	6.311	282
-30.1	6.600	305
-30.955	6.788	318
-33.1125	7.261	351
-34.3725	7.537	377
-35.99	7.892	402
-37.7575	8.279	436
-39.2425	8.605	459
-40.583	8.899	482
-42.1125	9.234	514
-43.7075	9.584	541
-45.4825	9.973	575

Sample-S-102		
Load (KN)	Stress (Mpa)	Strain Gage-1 (10 ⁻⁶)
0	0.000	0
-2.5575	0.561	10
-5.115	1.122	19
-7.6725	1.682	29
-8.715	1.911	39
-10.705	2.347	49
-12.1875	2.672	60
-14.05	3.081	77
-15.1225	3.316	86
-16.585	3.637	100
-18.18	3.987	111
-19.6425	4.307	126
-21.56	4.728	142
-22.3325	4.897	153
-23.6375	5.183	169
-25.055	5.494	186
-26.845	5.887	204
-27.7975	6.095	219
-29.2375	6.411	241
-30.4975	6.688	261
-31.78	6.969	282
-33.6	7.368	305
-34.455	7.555	318
-36.6125	8.028	351
-37.8725	8.305	377
-39.49	8.659	402
-41.2575	9.047	436
-43.2425	9.482	469
-45.1425	9.899	497

Sample-S-101		
Load (KN)	Stress (Mpa)	Strain Gage-1 (10 ⁻⁶)
0	0.000	0
-1.5575	0.342	9
-3.115	0.683	17.1
-4.1725	0.915	26.1
-5.215	1.144	35.1
-6.205	1.361	44.1
-7.1875	1.576	54
-8.55	1.875	69.3
-9.1225	2.000	77.4
-10.585	2.321	90
-11.18	2.452	99.9
-12.1425	2.663	113.4
-13.56	2.973	127.8
-14.3325	3.143	137.7
-15.1375	3.319	152.1
-16.555	3.630	167.4
-17.845	3.913	183.6
-19.2975	4.232	197.1
-20.7375	4.547	216.9
-21.9975	4.824	234.9
-23.28	5.105	253.8
-25.1	5.504	274.5
-25.955	5.691	286.2
-28.1125	6.165	315.9
-29.8725	6.550	339.3
-30.99	6.796	361.8
-33.2575	7.293	392.4
-35.2425	7.728	422.1
-36.6425	8.035	447.3
-38.6125	8.467	485.1
-40.2075	8.817	513.9
-42.9825	9.425	562.5
-45.2575	9.924	611.1

VITAE

SAMI S. HAMEED

CONTACT DETAILS

Mobile: +966 54 53 47 447

E-mail: samihmaid@hotmail.com

EDUCATION

M.S. degree in Civil Engineering, King Fahd University of Petroleum and Minerals (KFUPM).

B.S. degree in Architectural Engineering (Structural Engineering), King Fahd University of Petroleum and Minerals (KFUPM).

ACADEMIC EXPERIENCE

Graduate Assistant (Fall, 2010 & Spring, 2011)

Teacher assistant (Spring, 2008 & Fall, 2009)

NON-ACADEMIC EXPERIENCE

Projects Manager "Jan. 2014 - Present"

Structural Engineer "Oct. 2010 – Jan. 2014"

LANGUAGES

Bilingual (Arabic & English)